American Perfumer

This issue contains the eighth
in a *continuing* series of

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The aerosol documentary, consisting of
fifteen major articles, begins on page 37

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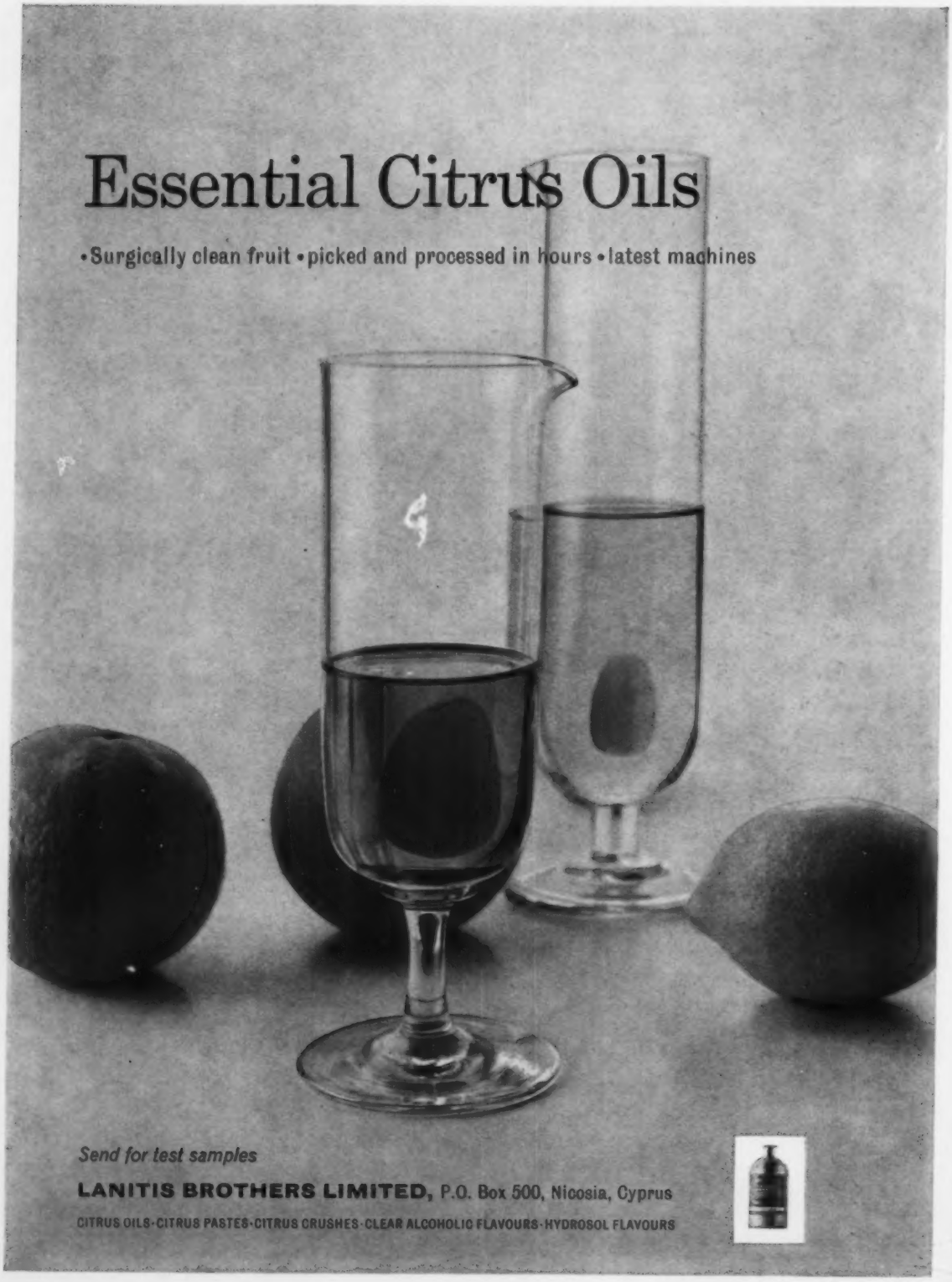

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American Perfumer

VOL. 75, NO. 10 OCTOBER, 1960

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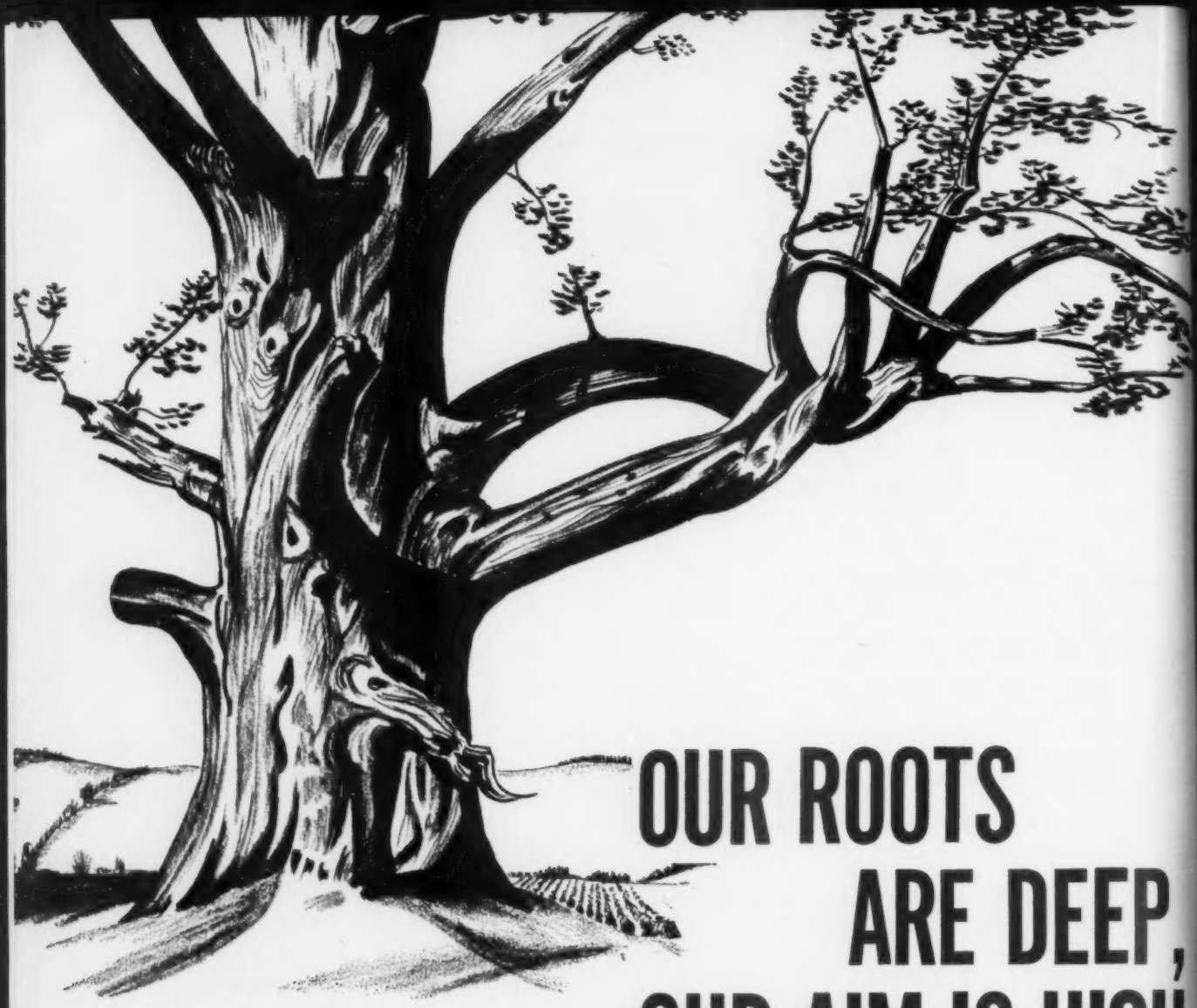

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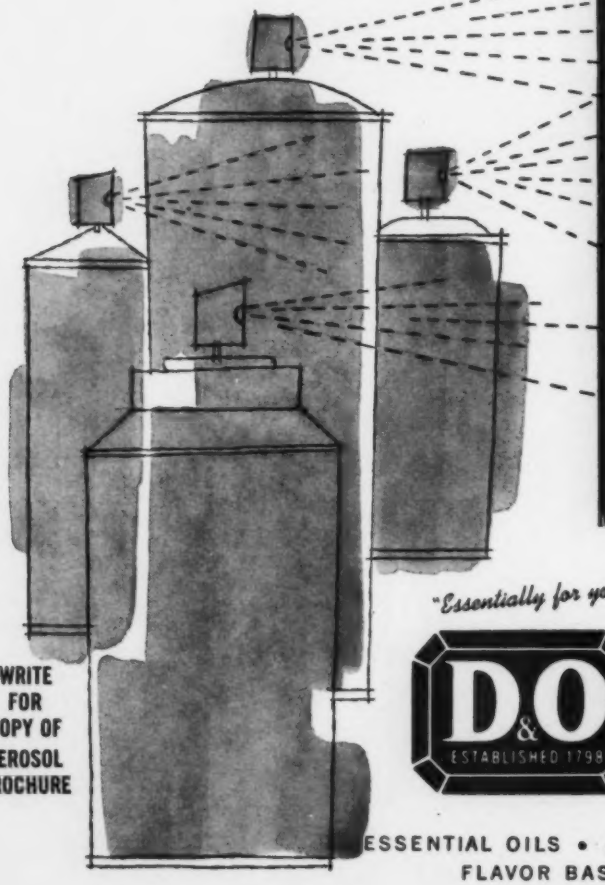

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Questions and Answers

Q. *We have been manufacturing a cold cream for a number of years. In the past we have put the cream into jars with a vacuum filling machine. Our method is to mix the cream until it cools down to 54°C., fill the jars and convey them through a refrigerated room to cool to room temperature before being cased and put in the warehouse. Our formula is the beeswax, borax type. We would like to cool this cream to approximately 45°C., fill the jars with a piston filler, put them directly into shipping cartons without passing them through the refrigerated room and let them cool to room temperature in the warehouse. We are concerned about the stability of our cream. Would it be apt to affect the quality of the cream using the latter method? Are there cold creams other than the beeswax borax type? J. L. P., California*

A. You do not really duplicate cold cream by making it out of anything else than beeswax, borax, mineral oil and water along with some possible modifiers. If you want to make a different kind of cream and to maintain a concentration of water such as you have in the present product, you can get rid of the odor of beeswax by not using it. We suggest that you try one of the Span-Tween formulas along this line and make it on the basis of holding about 30 per cent water which is probably what your cream contains. We believe it would be better for you to write to Atlas Powder Company, Wilmington 99, Delaware, and get their brochure on this subject. Self emulsifying glyceryl monostearate (such as Tegin) are useful too. Write to the Goldschmidt Chemical Corp., 153 Waverly Place, New York 14, N. Y. or the Kessler Chemical Co., Inc., 7292 State Rd., Philadelphia 35, Penna., for particulars on their products. Now as to temperature of filling and whether you should or should not run through your refrigerated room, rate of heat loss plays an important roll in the consistency, appearance and texture of the cream. It varies somewhat with the given formula. You might be able to fill your cream at 45°C., and put into shipping cartons and let them set in a refrigerated room or a cooled warehouse. We are afraid you are going to have to try this to see if it works. Generally, however, creams are filled at a somewhat lower temperature than what you are using. Off hand, a cream of your type would probably be filled in the vicinity of 32 to 35°C., at which point it starts setting fairly fast. Have you ever tried cooling your cream down to this temperature?

Q. *What is a dry skin cream? Can you give me a formula or refer me to a book? Also, what is a moisture cream, can you refer me to a book? S. A. Y., Belgium*

A. A dry skin cream is one that is intended for dry skins. It is generally more rich in emollients than the average skin cream. A moisturized cream is one that contains agents which hold or/and attract moisture to the skin. Among the moisturizing agents are the usual humectants, such as glycerol, butylene glycol, sorbitol syrup, the polyethylene glycols of molecular weight about 400 and similar products. Other ingredients, such as the unsaturates, lecithin and lanolin fractions all help to maintain moisture in the skin. We suggest that you also look into the book by Sagarin "Cosmetics: Science and Technology" or Harry's recent book on "Modern Cosmeticology."

Q. *We are interested in manufacturing a bleaching cream of the vanishing type using hydroquinone. Can you advise us in regard to formulation? C. S. M., Virginia*

A. We do not recommend the manufacture of bleaching creams containing hydroquinone. Indeed, hydroquinone was used in a concentration of 0.1 per cent as an antioxidant in a baby oil some years ago and was withdrawn from the market. On the other hand, hydroquinone is mentioned in a sun screen patent used in combination with ascorbic acid. At one time a commercial product was reputed to have this composition. Whether this is any longer the case, we are unable to say. In summary, hydroquinone is not considered to be a particularly safe material to apply in any concentration to the skin. While it may be used, we are skeptical of its skin safety.

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DR. FRED T. REED is a native of Wilmington, Delaware. He was graduated from Swarthmore College with a bachelor's degree in chemistry, then joined the Army. He served in the Pacific area during World War II, attaining the rank of captain. Following his discharge from the Army, Fred entered the University of Maryland, where he obtained his Ph.D. degree in chemistry. He joined the Du Pont Company as a research chemist in 1950 and was assigned to the "Freon" Products Division. Fred has done a variety of work for the aerosol industry including methods for moisture analysis of propellents, propellent systems for low-pressure cosmetic aerosols and studies of anti-perspirant compounds. Currently, Fred is in charge of all sales service work for the aerosol industry at the "Freon" Products Laboratory at Chestnut Run near Wilmington.

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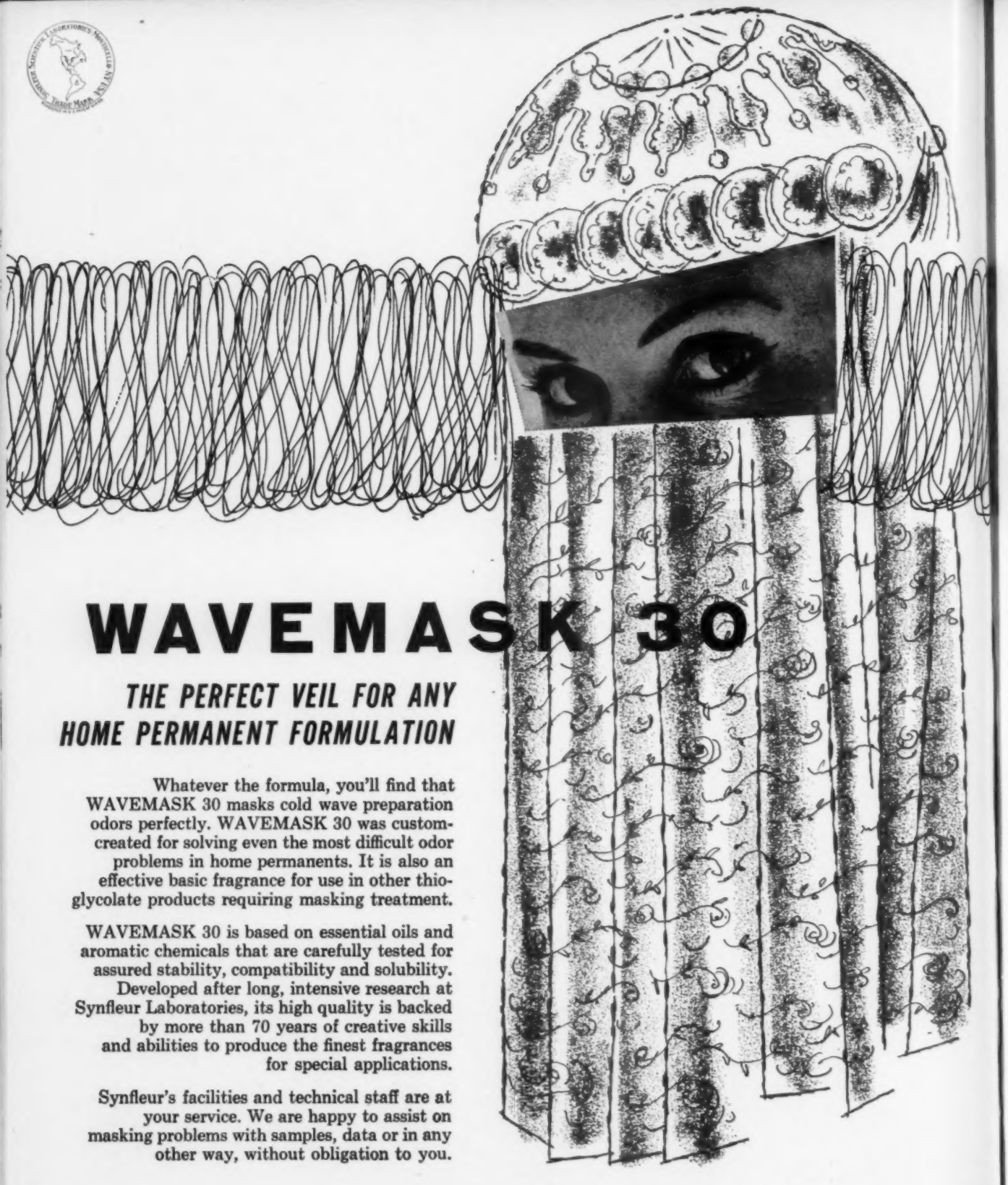
*** Michel Prunieras, LA THERAPEUTIQUE TISSULAIRE EN DERMATOLOGIE, No. 205, July 1949**



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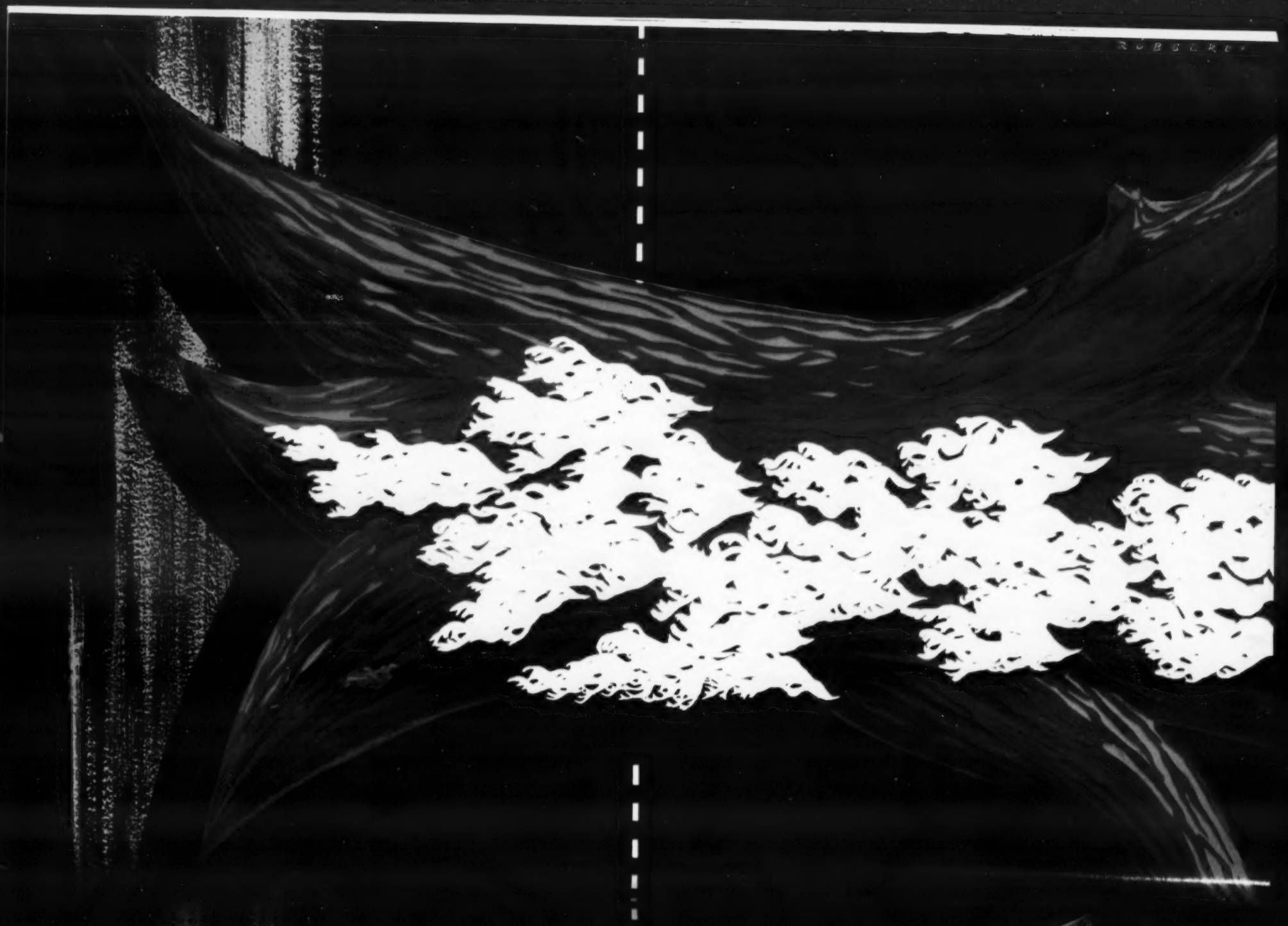

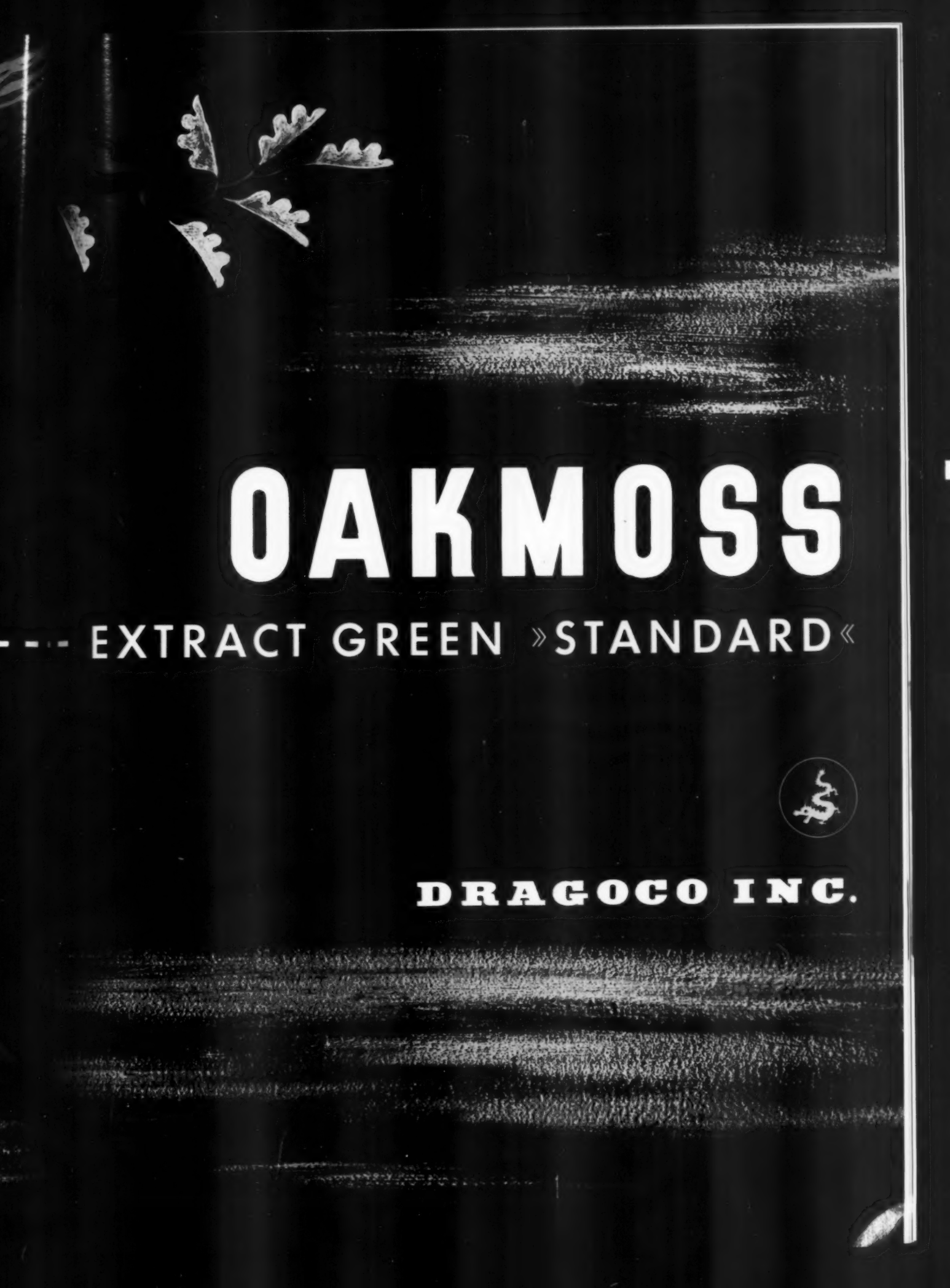
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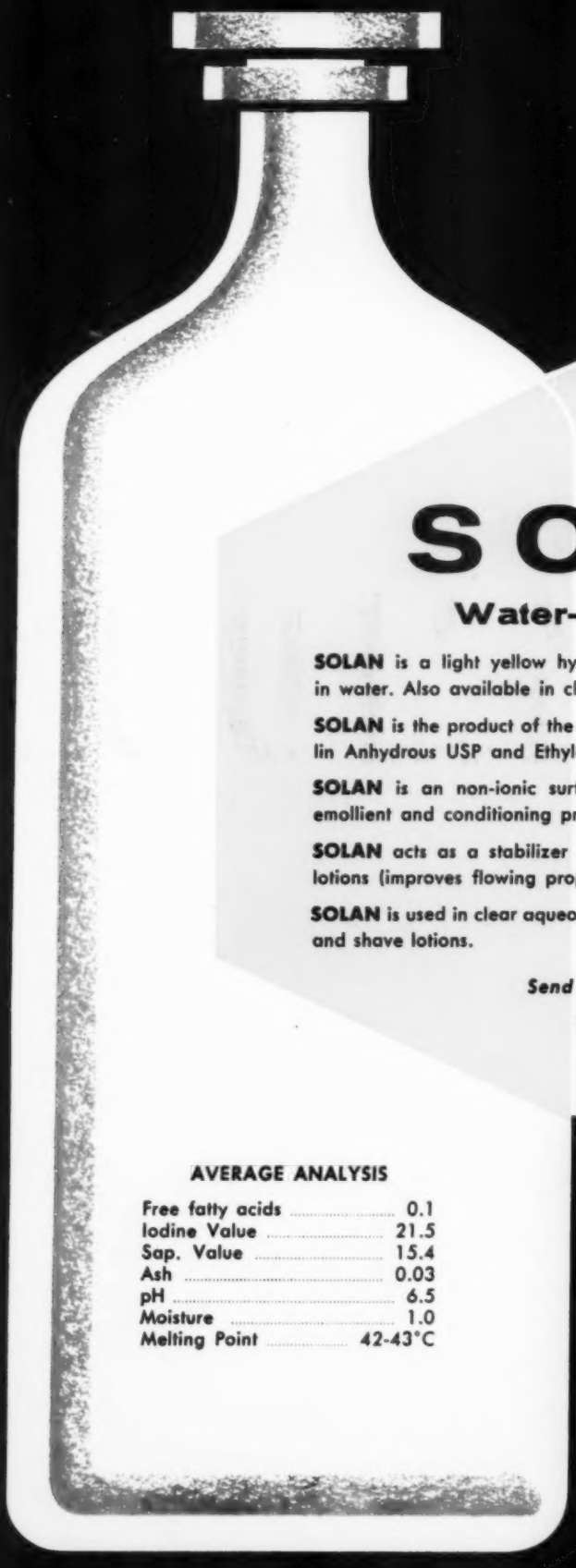

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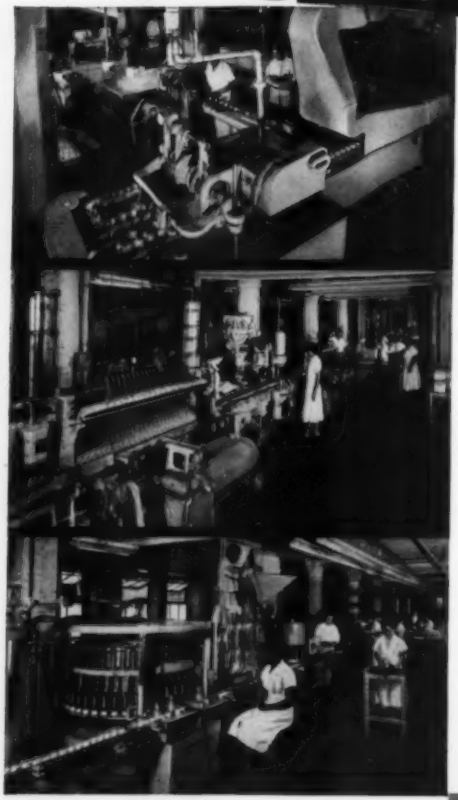

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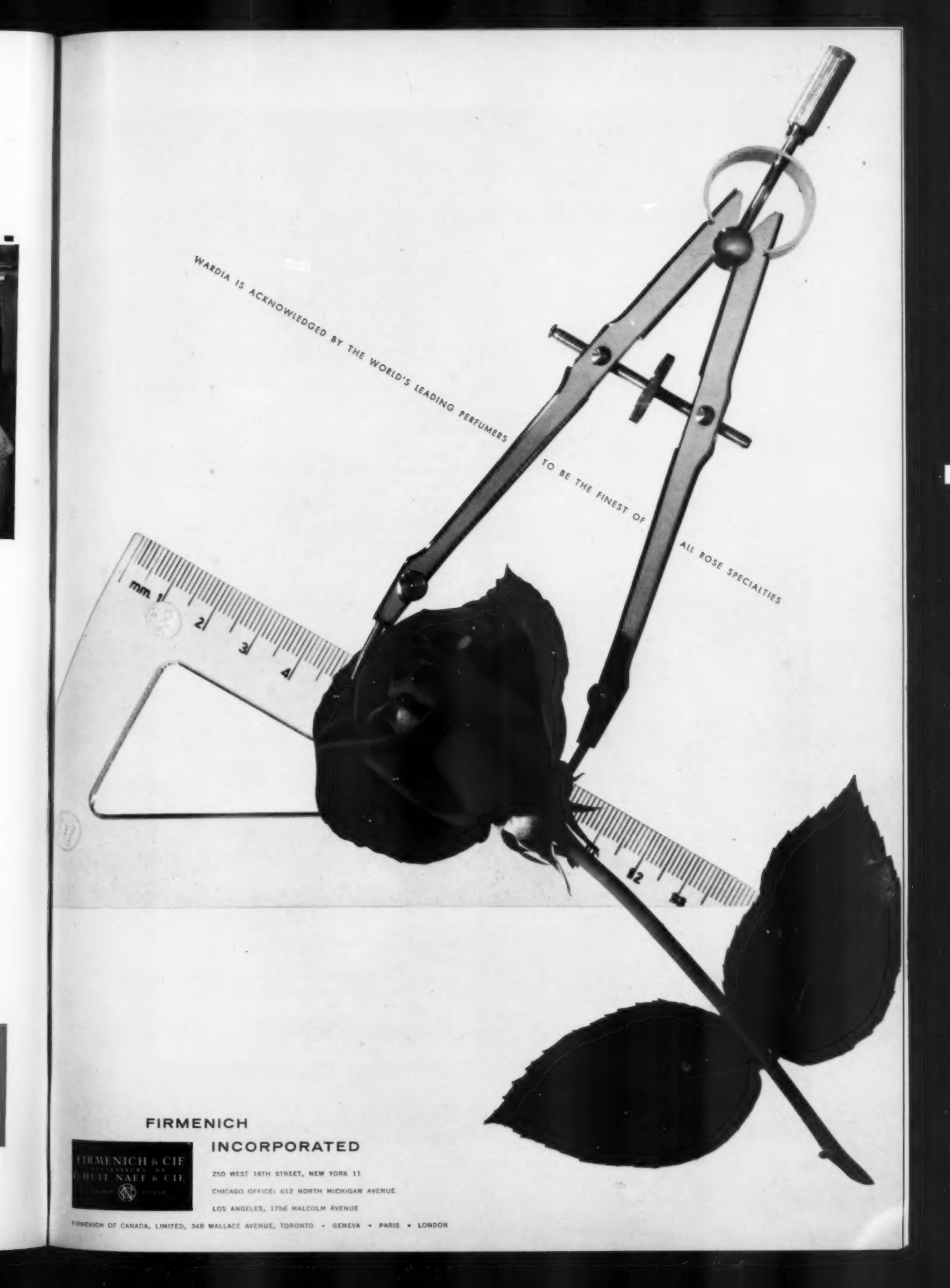
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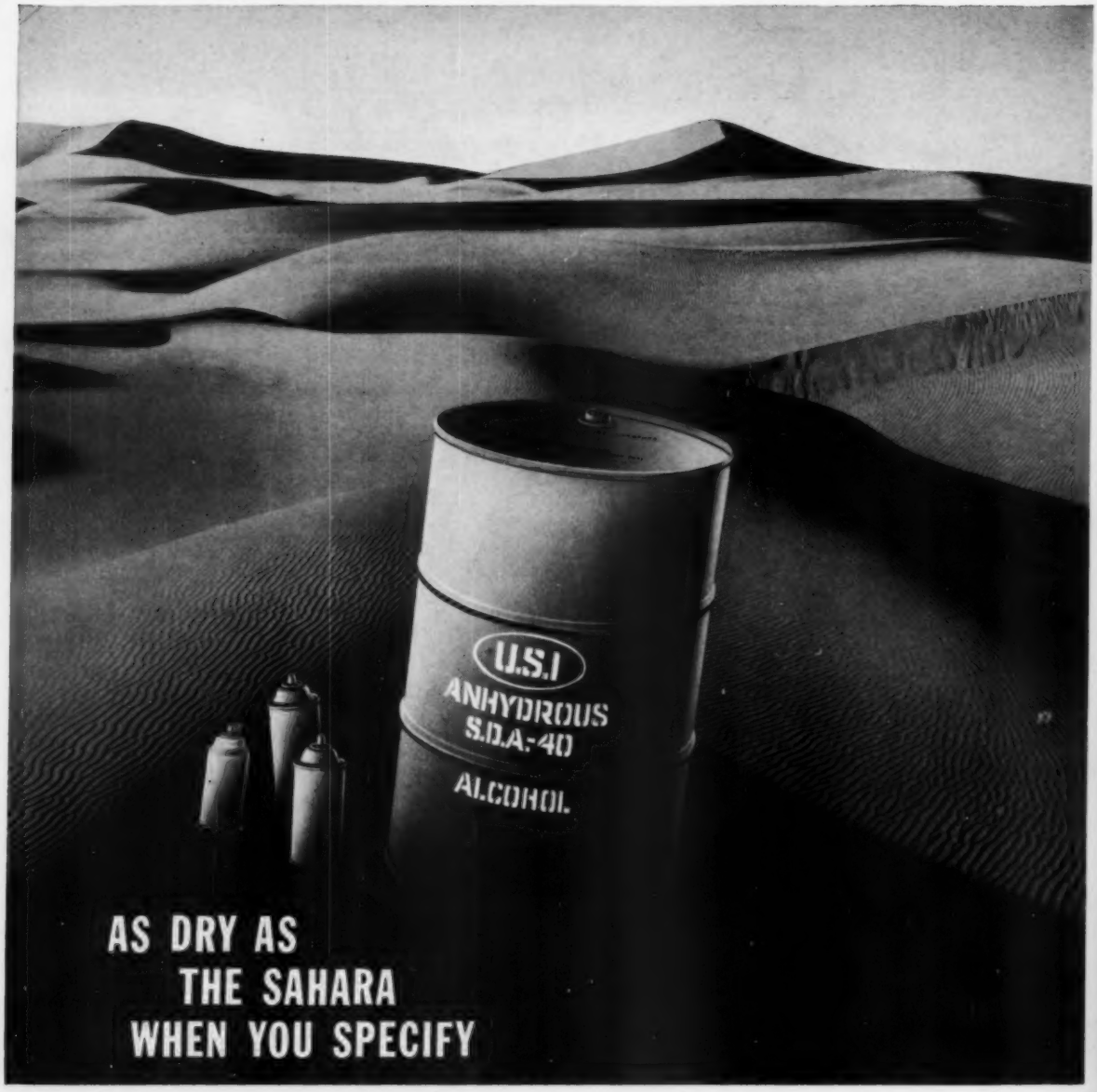

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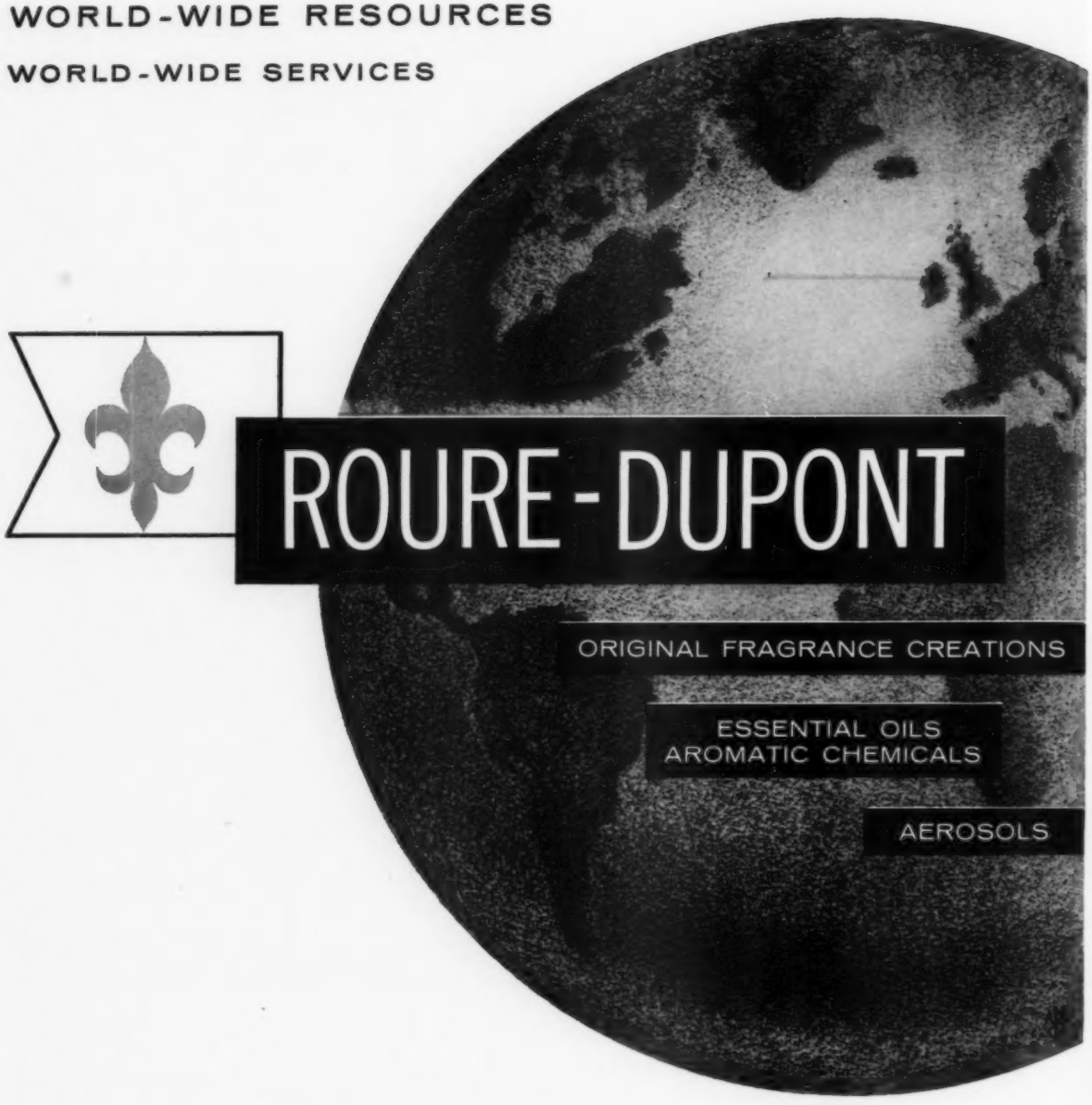

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Notes

Glycol ethers of cetyl, stearyl and behenyl alcohols have been patented, Indian Pat. #70670, as superior in depressing water evaporation at ambient temperature ranges 10-35°C . . . . . According to the grapvine, Joe Kalish and Marvin Cook are preparing a book on Cosmetics. Good Luck! . . . . . It is interesting to note that profits after taxes in the drug industry for 1959 are around 11% while the cosmetic industry shows only 6.5% . . . . . Was happy to see Bob Marriott chosen as the S.C.C. medalist for 1960. He is also president 1960-61 of the I.F.S.C.C. . . . . Salt is the basis of a new British tooth paste . . . . . A buffered solution of sodium propionate is useful as an eye drop in treating eye infections . . . . . Azulene is suggested for use in powders for treating dermatoses, diaper rash, heat rash and various types of pruritis . . . . . Good luck and congratulations to Veronica Conley who is now Director of the new Department of Nursing at the A.M.A. . . . . Phenyl nitroethane smells like cinnamon and is found in one of the varieties of Ocotea Pretiosa according to Gottlieb and Magalhaes . . . . . Givaudan have just introduced an interesting new polycyclic alcohol that reproduces the odor of sandalwood (with vetivert overtones in my opinion) at less than half the cost of sandalwood oil. It can be used in soaps, perfumery, creams and lotions . . . . . If there was ever any doubt of the political interests of H.E.W. secretary Fleming, his recent remarks at a press conference put the record straight . . . . . Big new breakthrough in anticaries agents will be an additive to sugar which prevents acid formation and consequent tooth decay—You have guessed it; someone is working on it . . . . . Dow have at least two patents on oxalkylation of hydroxy compounds (BP 831,900 and 832,-601) such as sugars and glycerol . . . . .

Justin Powers

The pharmaceutical scene loses the services of an outstanding man in the retirement of Justin L. Powers, Editor of the A.Ph.A. Journal, Scientific Section and revision director of the National Formulary. He has also been active on the U. S.P., the World Health Organization; recently he was given the Remington Medal.

Justin Powers first came to my attention in 1940 during the preparation of a chapter on emulsifiers, through his paper, coauthored by Leask and Warner, when he was at the University of Michigan, College of Pharmacy. Then as now he was all help and cooperation.

Although deserving of retirement after 25 years of serving the membership of the A.Ph.A., Dr. Powers will undoubtedly not spend his days sleeping and otherwise resting. So, to his "retirement", may it be as rewarding as his "active" days; let sunshine and good health be with him always.

Placenta Facts

Some readers will recall my questioning of the morning speakers at the S.C.C. Seminar in Chicago on September 15th regarding placenta extract and its possible effects on skin.

I don't think the answers given can be taken as presented. Just because we don't know how to

For information, write Dept. AP-10

measure the effectiveness of a new ingredient doesn't mean it is worthless. This goes for royal jelly and anything else.

Placenta extract *does* increase skin respiration of animal skin. A paper will soon appear in the American Perfumer and Aromatics on this. It will probably have a similar effect on human skin.

Most cosmetic materials do depress skin respiration. And so, if the addition of placenta extract does increase the rate of skin respiration at all, thereby neutralizing the depressing effect of other ingredients, it has a place in the armamentarium of cosmetic materials. Indeed, the remark made by one of the experts that morning indicated that placenta extract had no influence on increasing skin respiration, based on his tests. Yet I remember a statement made in connection with a test using a preparation containing placenta extract which said in part that it had a "slight, but definite, stimulating effect (on skin respiration) in that it tends to elevate the rate of cutaneous respiration . . ." (my parenthesis).

Only in the past year or so, has any serious scientific work been done to determine methods of standardization of placenta extracts some of which was reported before the S.C.C. meeting last May and again at the I.F.S.C.C. Congress in Munich, on September 1st, 1960. Among the serious work done was the determination of the effect of preservatives, antioxidants, pH, temperature and other factors.

Placenta extract may well be one of the most useful materials for dermatocosmetics offered in the last 50 years or longer. Don't sell it short until all the facts are in.

Novel Letter

The following letter was recently received by a customer from one of his suppliers:

"Gentlemen:

"This letter is different, we hope, than most of the correspondence you receive, for in this letter we are saying

THANK YOU.

"You are a good customer with a very satisfactory payment record and one of the very valuable assets of our business. All of us in the Credit and Collection Department want you to know that your excellent payment record makes our life more pleasant and we also express our appreciation for your patronage.

"Your business in the past is sincerely appreciated and it is our hope that we may continue to supply our quality products to you for many years.

Cordially,"

How do you like that for public relations?

Citizen versus physician and vitamins

The August 20th issue of the Journal of the A.M.A. has an editorial entitled "Vitamins-Good or Bad". In it the statement is made that ". . . . . it is the responsibility of the physician to recommend supplementary vitamins and the decision regarding dosage and kind of vitamins must reside in the physician and not in the patient or public press."

All this in regard to taking vitamins on your own hook without consulting a physician.

As the article points out the average diet in the U.S.A. is pretty well covered by vitamins. But it is equally true that borderline deficiencies can and do exist. Can the physician recognize them? Maybe. In general, he is just as guilty of misusing multiple vitamin preparations as the citizen. Worse yet, the average citizen takes "weak" stuff compared to the far more potent preparations prescribed for him by his physician.

The number of actually serious deficiencies requiring high potency medication would hardly keep one company in business and would not necessitate the wide advertising of these products in medical and pharmaceutical journals.

So, lets not leave the implication that the citizen, manufacturer or vendor of food supplements are the culprits. The real fault may be with the physician indiscriminately prescribing expensive, overly potent preparations.

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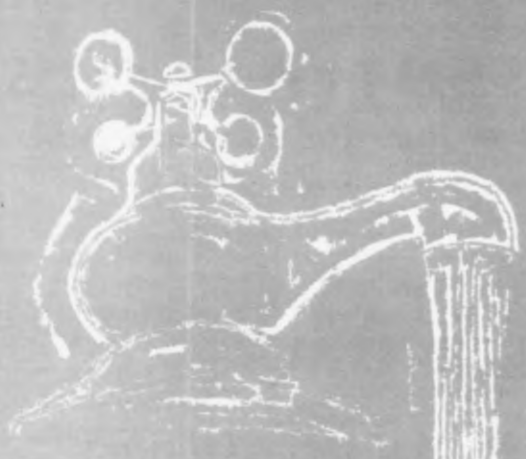

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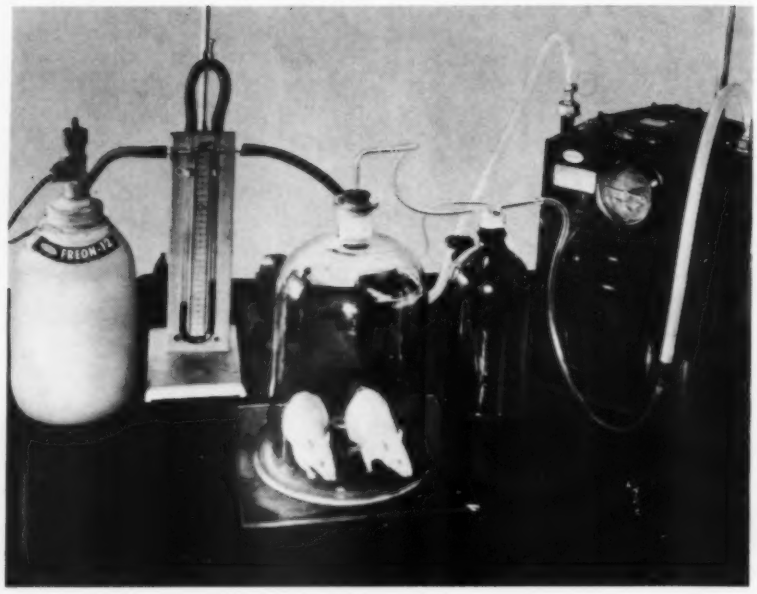

Test animals being exposed to propellent gas. The propellent is lead through a flow-meter into a mixing chamber. Here, 20% by volume is mixed with a measured amount of air, and the whole run through a test chamber with the animals.

Toxicity of Propellents

By Fred T. Reed
E. I. du Pont de Nemours & Co.

Of the many property requirements which an aerosol propellent must meet, certainly low toxicity heads the list. Vapor pressure, boiling point, solvent power and flammability are critically important properties, but even if these properties were ideal, a propellent material would be completely unacceptable if it did not have a low order of toxicity. In the discussion which follows only those propellents are considered which meet this rather broad requirement of low-order toxicity.

The primary concern in considering toxicity of aerosol propellents is inhalation. In addition, certain other areas are of interest, such as ingestion toxicity, skin sensitization and toxicity of decomposition products. In the last case, this usually is important only as thermal decomposition, as would occur when an aerosol product is sprayed into an open flame. When chemical decomposition of the propellent occurs in the container, the product is normally unacceptable for its intended application and thus would not be marketed. Of the various types of toxicity mentioned, only inhalation toxicity has been extensively studied for the many compounds which are used as propellents. The most detailed information available covers the fluorinated hydrocarbon propellents (Ref. 1).

The Underwriters' Laboratories have investigated several of the fluorinated hydrocarbons. Their findings are reported as group classifications. Tables II and III give these group classifications for all the common propellents and some of the less common propellents. Table II is intended as a reference table to give some idea of the types of compounds which

would fall into the very low (more toxic) Underwriters' Laboratories classifications. Common materials with which the reader would normally be familiar were selected and, as is obvious, these materials listed in Table II would be completely unacceptable as propellents. In the case of methyl chloride, CH_3Cl, special handling conditions would be necessary because of its toxicity if it were used as a propellent.

The definition of the Underwriters' Laboratories classes are given in Table I. It is well to remind the reader at this point that this classification is based on inhalation toxicity only. Further details on this classification can be found in Ref. 1 and the references contained therein. Where no information is available a blank space is indicated.

Probably the most interesting point to note in Table III is the fact that Propellents 12, 114, 114a, 152 and C318 are grouped in the highest classification and indeed are less toxic than carbon dioxide. The latter normally constitutes a substantial percentage of the gases in the lungs which makes it quite apparent that those materials classified in Group 6 would be completely free of any inhalation hazards in their normal use as propellents. It is also worth noting that most of the compounds listed in Table III fall in a classification as high as carbon dioxide. Those few which fall below carbon dioxide, such as methylene chloride, still present no hazard in the quantities which are normally used in an aerosol product and in the manner in which the consumer uses the product.

It would be advisable at this point to remind the reader that none of these compounds will support life. Thus, should any situation occur where the oxygen content of the atmosphere reaches such a low level as to fail to support life, then any of these compounds, if it is the only other gas present, becomes a serious hazard. This is a somewhat cumbersome way of saying that any of these propellents will cause suffocation if insufficient air is present. This becomes a problem only to the manufacturers of propellents and loaders of aerosol products. One might visualize the situation where a workman is asked to clean a tank which previously contained one of these propellent materials or possibly a solution of materials, including one of these propellents. The tank will certainly be filled with the vapors of the propellent and it is very likely that essentially no air will be in the tank. Unless the tank is well ventilated or the worker is wearing an air mask (not a gas mask), he will surely suffocate.

In addition to the Underwriters' Laboratories classifications a very useful tool in determining the inhalation toxicity of the vapors of a liquid is to refer to the "Maximum Allowable Concentration" (M.A.C.) published in various references on safety. An excellent source of listings of M.A.C. values are those given by the American Conference of Governmental Industrial Hygienists. These values are expressed as parts per million by volume of gas in air. It should further be explained that M.A.C. values are those levels of vapors which are harmless to humans under conditions of continued daily exposure year in and year out. Thus, they would be levels at which workers could be exposed to these gases throughout their working life time without injury. M.A.C. values are listed in Tables II and III for those compounds where the values have been published and a reasonable degree of accuracy indicated. Values listed in parentheses are extrapolated values. In the cases where this occurs in Table III the values are undoubtedly correct. The reader will note that none of the M.A.C. values listed exceed 1,000 ppm except carbon dioxide. It has been the practice in reporting M.A.C. values that no compound be listed with a value exceeding 1,000. Carbon dioxide is a special case since it is a component of exhaled breath. As might be expected, some discrepancies occur between the Underwriters' Laboratories classifications and the M.A.C. values. From a practical standpoint this is of no importance to the marketing of aerosol products. It is quite unlikely that anyone would consider using a propellent whose M.A.C. value is less than 100. Such a propellent would certainly place it in an Underwriters' Laboratories class of four or less.

In addition to inhalation toxicity, ingestion toxicity is becoming of greater interest to the aerosol field. Food products are already being marketed and many new ones contemplated. Included in Table III is a tabulation of approval by the Food and Drug Administration for the compounds listed. As yet, none of the halogenated compounds have been approved. The hydrocarbons, propane and butane, and the compressed gas propellents, carbon dioxide, nitrous oxide, nitrogen and air have all been approved. While the latter two compounds have not been specifically mentioned in any FDA listing, the writer assumes that there would be no question of their use since these gases normally occur in all food products.

To the writer's knowledge, the only halogenated compound for which FDA approval is being sought is "Freon-C318". The Du Pont Company has submitted data to the Food and Drug Administration covering ingestion toxicity studies with this compound and approval is expected by mid-1960.

The most difficult problem to overcome in obtaining acceptance of the commonly used propellent materials, namely the fluorinated hydrocarbons, is the generation of fluoride ion through hydrolysis on long term storage of aerosol food products with these propellents. While no acceptable tolerance level for fluoride ion has been stated by the Food and Drug Administration, it is almost certain that the generation of more than one or two ppm of fluoride ion per year would be unacceptable. This is an extremely difficult requirement to meet and it is very unlikely that any of the fluorinated hydrocarbons now being used by the aerosol industry could meet this requirement, even though some of them, such as Propellent 114, exhibit very low hydrolysis rates even in the presence of the acidic medium found in some food products.

Little data are available on skin sensitization of the commonly used propellent materials. However, the writer is aware of no cases of skin sensitization caused by propellents despite the tremendous exposure through the use of aerosol personal products. An obvious reason for this lack of data is the inability to conduct such tests because these materials are so volatile. Rabbit-eye tests by the Du Pont Com-

pany (Ref. 1a) have shown that the Propellents 12, 11 and 114 cause no reaction. These tests were conducted using a solution of the propellent in mineral oil at atmospheric pressure. Because of the low solubility of the propellents at atmospheric pressure, the concentration of propellent in the test solution was necessarily low. Nonetheless, this would represent realistic conditions.

All of the liquefied propellents are potential refrigerants and as the reader is aware, some of them are widely used as commercial refrigerants. This fact raises an important precaution which should be considered in handling and marketing aerosol products. If a workman or the consumer is sprayed by gross quantities of liquid propellent, freezing of the skin is a definite possibility. This is more often a problem with the workman than with the consumer since the consumer has considerable control of the aerosol as it is applied. Should he tend to spray an excessive quantity of the aerosol on his skin, he would be given adequate warning by the immediate chilling affect. It would certainly be wise, however, for the marketer of personal products to include on the label a warning against excessive spraying, with particular emphasis on spraying in the eyes.

A final problem concerned with the toxicity of aerosol propellents is that arising from the decomposition of the halogenated propellents in an open flame or on exposure to very hot surfaces. Considerable information on this problem can be obtained in Ref. 1. The main products of thermal decomposition from the halogenated propellents are halogen acids, carbon dioxide and traces of phosgene. Phosgene is quickly hydrolyzed to carbon dioxide and hydrochloric acid. The literature indicates that the toxicity of phosgene and the halogen acids are essentially equivalent. Phosgene, probably because it was used as a war gas, has often been stated as the real danger of decomposing halogenated propellents. This is not true. The halogen acids are equally as dangerous and are present in considerably greater quantities. Fortunately, thermal decomposition is not a serious problem. The halogen acids are so irritating that, even at concentrations below any seriously toxic level, a person normally cannot remain in such an atmosphere. For example, should a housewife spray an aerosol insecticide directly into an open gas flame in her kitchen for a sufficient period of time to generate quantities of decomposition products, she would be driven from the room immediately by the irritating effects of the halogen acids. If the reader is interested in exploring this problem in greater detail, he is referred to Ref. 1 and the references contained therein.

In summary, it appears that the commonly used propellent materials present no hazard from inhalation. As a rough guide, those compounds falling in Underwriters' Laboratories classification of 5 and 6 or having M.A.C. values above 500 appear to be perfectly safe to use. Borderline conditions probably occur at Underwriters' Laboratories class 4 and M.A.C. values of 100-500. Any materials rated below these values would undoubtedly be unacceptable. For aerosol products which are to be ingested, it would appear that the formulator is limited to the compressed gases indicated in Table III. The remaining two general areas of toxicity, skin sensitization and thermal decomposition products, do not appear to be serious problems.

Table I

Underwriters' Laboratories Classification	Definition
1	0.5 to 1 vol. % serious injury in 5 minutes
2	0.5 to 1 vol. % serious injury in 30 minutes
3	2 to 2.5 vol. % serious injury in 1 hour
4	2 to 2.5 vol. % serious injury in 2 hours
4-5	Less toxic than 4 and more toxic than 5
5a	Much less toxic than 4 but more toxic than 6
5b	Data indicate classing as 5a or 6
6	20 vol. % no injury in 2 hours

Table II

Compound	Underwriters' Laboratories Classification	Maximum Allowable Concentration
SO_2	1	10 ppm
NH_3	2	100
CCl_4	3	25
CH_3Cl	4	100

Table III

Propellent	Underwriters' Laboratories Classification	Maximum Allowable Concentration	Food and Drug Administration Approval
11, CCl_3F	5a	1,000 ppm	No
12, CCl_2F_2	6	1,000	No
21, $CHCl_2F$	5	1,000	No
22, $CHClF_2$	5a	1,000	No
30, CH_2Cl_2	4-5	500	No
113, $CClF_2CCl_2F$	4-5	(1,000)	No
114, $CClF_2CClF_2$	6	1,000	No
114a, CCl_2FCF_3	6	(1,000)	No
142b, CH_3CClF_2	5a	....	No
152a, CH_3CHF_2	6	....	No
160, CH_3CH_2Cl	4-5	1,000	No
1140, $CH_2{=}CHCl$	(6)	500	No
C318, $CF_2CF_2CF_2CF_2$	6	(1,000)	No
CO_2	5a	5,000	Yes
N_2O	..	....	(Yes)
N_2	6	....	(Yes)
Air	6	....	Yes
Propane	5b	(1,000)	Yes
n-Butane	5b	(1,000)	Yes
i-Butane	5b	(1,000)	Yes

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Hair sprays—Past, Present, Future

By M. J. Root
G. Barr & Company

Although the first hair fixatives that were used may have been the mud from the banks of the River Nile, it was probably not long before it was discovered that the secretion of the lac insect dissolved in spirits made a far more utilitarian product. And so, up until about 1950 hair fixatives utilized such natural-occurring resins as shellac, benzoin, and rosin dissolved in ethyl alcohol. These products were applied to the hair by means of an atomizer, nebulizer, or squeeze bottle and then usage was confined almost solely to the beauty shops. The function of these products was to hold the hair style in place and thereby give it a "sculptured look." Acceptance of these products by the general public awaited a better means of application, which was provided in 1948 by the appearance of the low-pressure aerosol container. Aerosol hair sprays comprise a resin, plasticizer, additive, solvent, perfume, and propellent.

There are several requirements for a resin to make it suitable for use in a hair spray, which are as follows:

1. Soluble in anhydrous ethyl alcohol and not precipitate out at low temperatures required for cold filling of the product;
2. Soluble in the ethyl alcohol-propellent system;
3. Functional groups with affinity for the functional groups in the hair, such as the carboxyl, amino, hydroxyl, and amide;
4. Low water absorption so that the curl will be retained at high humidity;
5. Easy removal from the hair with water and soap;
6. Clear film that can be suitably plasticized;
7. Reasonable cost. The price of the monomer used is the preponderant factor determining the cost;
8. Availability. The resin must be commercially manufactured.

The total resin content of aerosol hair sprays on the market varies from 1% to 2-1/2%.

Shellac

Since shellac had been used for many years as a hair fixative, it was natural that the first aerosol hair sprays contained shellac for the resin. It very quickly became obvious that the impure yellow shellac containing large amounts of water which had been used in hair fixatives was not desirable for use in aerosol hair sprays. As a consequence, very soon bleached, low-wax and moisture shellac was specified for use in pressurized packages. It was also very quickly realized that shellac had a serious shortcoming, in that it is not soluble in water and hence removed from the hair with difficulty. Since shellac is a mixture of polyhydroxy acids, it can be water solubilized by neutralizing with triethanolamine or ethoxylating. Also, it can be made dispersible in water by the addition of a surface active agent. As early as 1950, a water soluble ethoxylated shellac (Elastolac) hair spray was being marketed.

Another drawback of shellac is its corrosive effect on tinplate containers, which is the most commonly used container for hair sprays. The use of esterified shellac has overcome this problem to a large degree.

Polyvinyl pyrrolidone

Reppe's high-pressure synthesis in 1930 of vinyl pyrrolidone made available polyvinyl pyrrolidone which was first utilized as a blood plasma extender. This resin was first manufactured in Germany during World War II and its manufacture in this country was taken over by General Aniline & Film Corporation. To sustain the demand, research was intensified to discover other uses, and as early as 1950 its application in cosmetic products was being tested. Because of its solubility in water and anhydrous alcohol, its suitability for use in hair sprays was quickly recognized. In 1953 the first aerosol hair spray products with polyvinyl pyrrolidone as the resin appeared on the market.[1]

Polyvinyl pyrrolidone is available in three viscosity grades, K-30, K-60, and K-90, but the lowest viscosity grade, K-30, is the one generally used in hair sprays.

Since polyvinyl pyrrolidone is a hygroscopic material, films of the resin become tacky under conditions of high humidity. At 50% relative humidity, the equilibrium water content of an unmodified film is 18%.

An attempt to reduce the hygroscopicity of polyvinyl pyrrolidone by the addition of shellac proved disastrous. The shellac reacted with the polyvinyl pyrrolidone to form a residue which was insoluble in the alcohol-propellent system that clogged the valves.

Vinyl pyrrolidone/vinyl acetate copolymers

Vinyl pyrrolidone can be polymerized with varying percentages of vinyl acetate to give copolymers especially useful for aerosol hair sprays. The resin known as PVP/VA E735 is a copolymer containing a ratio of 70/30 vinyl pyrrolidone/vinyl acetate, and is available from General Aniline & Film Corporation in a 50% solution in SDA-40 anhydrous ethanol. Resins are also available in different ratios of vinyl pyrrolidone/vinyl acetate such as 60/40, 50/50, and 30/70. The hygroscopicity of the PVP/VA copolymers is a function of the vinyl pyrrolidone content; the greater the vinyl pyrrolidone, the greater the hygroscopicity.

Vinyl pyrrolidone can be polymerized with other monomers, but none that has been investigated appears to be useful for hair sprays.

Hair sprays made with PVP/VA E735 resin form clear, lustrous films which are water soluble, yet not tacky at high humidities. The water solubility allows easy removability of the hair spray, as well as resetting of the hair with a wet comb.

Modified acrylic resins

Base 325, marketed by Ciba Company, Inc., is another resin that is used in aerosol hair sprays. This resin is a neutralized polyethyl acrylate. It yields clear, lustrous films which are only slightly soluble in water. It is easily removed from the hair with warm water. This resin is presumably covered by Ciba's British Patents #796,318 and #796,319.

Quaternized acrylic resins

A quaternized resin, such as a terpolymer comprising diethylaminoethylmethacrylate, methyl methacrylate, and lauryl methacrylate, quaternized with dimethyl sulphate is also being used in hair sprays. This resin was available in experimental quantities for a short time from duPont under the designation TLF-1218-B. A modification of this resin is now being manufactured by a hair spray marketer for their own use.

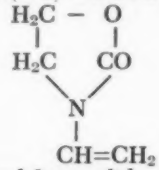
Dimethyl hydantoin formaldehyde resin DMHF

This resin, which is soluble in both water and anhydrous alcohol, has also been found useful for hair spray formulations. It was first made available by duPont and now is marketed by Glyco Products, Inc. The resin is manufactured by reacting dimethyl hydantoin with formaldehyde. Care must be taken to remove all the free formaldehyde when the reaction is completed. This can be done by reacting the formaldehyde with urea, amide, or sulfites.

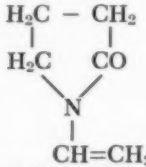
This resin forms clear, brilliant films by spraying of its alcoholic solution. The film is readily removed with shampoo and the hair can be reset with a wet comb.

N-vinyl-5-methyl-2-oxazolidone-vinyl acetate copolymer

Some time ago American Cyanamide prepared by a new synthesis N-vinyl-2-oxazolidone. The structural similarity between this compound and N-vinyl-2-pyrrolidone (VP) is shown below:



N-Vinyl-2-oxazolidone
(cyclic carbamate)



N-Vinyl-2-pyrrolidone
(cyclic amide)

Homopolymers of N-vinyl-2-oxazolidone—like vinyl pyrrolidone—are water soluble.

The Dow Chemical Company carried on the development with these monomers and made available ET-286[2] (Devlex 130) which is a poly-N-vinyl-5-ethyl-2-oxazolidone. Later the copolymer with vinyl acetate was made available under the designation ET-358 (Devlex A-515). This copolymer does not have the solvency in alcohol-propellent mixtures that the vinyl pyrrolidone-vinyl acetate copolymers have and therefore co-solvents such as methyl chloroform (1,1,1-trichloroethane) and methylene chloride must be used.

Carboxylated copolymers of vinyl acetate

The introduction of carboxyl groups into vinyl acetate polymers which can be neutralized solubilizes these resins in alcohol and water. Two such resins have been recommended for use in hair sprays.

Gelva C-3V-10R, which is produced by Shawinigan Resins Corporation, can be neutralized with triethanolamine to solubilize the resin in absolute alcohol provided an auxiliary solvent such as methyl chloroform is used also.

National Starch Products, Inc. has recently made available a resin which is also a carboxylated polyvinyl acetate designated Resyn® 28-1310. This resin can be neutralized with various organic amines to produce solubility in alcohol and water. The percentage neutralization can be varied to give hair sprays which differ in their degree of softness and water sensitivity. For good water solubility of the film National Starch recommends that the resin be 90% neutralized with 2-amino-2-methyl-1,3-propanediol (AM PD).

Other amines can be used to neutralize Resyn® 28-1310, with varying solubility and film characteristics. The following table gives the film characteristics with various organic amines:

Amine	Film Characteristic
Triethylamine	Cloudy film — non-tacky
Diisopropylamine	Cloudy film — non-tacky
Di-2-ethylhexylamine	Cloudy film — non-tacky
Tris(hydroxymethyl)amine-methane	Cloudy film — slightly tacky
2-Amino-2-methyl-1-propanol	Cloudy film — non-tacky
Tertiary-butylamine	Clear film — non-tacky
Monoisopropanolamine	Clear film — slight tack
Isopropylaminoethanol	Cloudy film — non-tacky
Di-n-butylamine	Cloudy film — non-tacky

In all the above instances a 5% solution of the resin in SDA #40 anhydrous alcohol was used and sufficient amine was added to obtain a pH of 8. Film characteristics were evaluated by spraying onto a watch glass.

Plasticizers

The list of plasticizers that have been used in aerosol hair sprays is quite long. Plasticizers can perform the following functions in a hair spray:

1. Harden or soften the film
2. Reduce or increase tack
3. Modify film clarity
4. Increase or decrease hygroscopicity.

Generally, the plasticizer is compatible with the resin, but this need not always be the case. Plasticizers such as silicones can be used which are not compatible with the resin. In this case the function is not really plasticization, since the film characteristics are not affected per se, but rather the surface.

Following is a partial list of plasticizers that have been used with various resins in hair spray formulations:

Castor Oil
Polyethylene glycol dilaurate
Lanolin
Dow Corning Silicone DC555
Polyethylene Glycol 400
Dimethyl Phthalate
Myristyl alcohol
Citroflex A-2 (acetyl triethyl citrate)
Lanolin esters
Lanolin alcohols
Cetyl alcohol
Urea

As can be seen, the list includes solids, liquids, and pastes. Many other materials have been used for plasticization of the resins in hair sprays; sometimes even a combination of resins can be used to give specific film properties. Thus, for example Shawinigan recommended the use of dimethyl hydantoin formaldehyde resin (DMHF) to reduce the stiffness of their Gelva C-3V-10R resin.

Additives

Various additives have been used in aerosol hair sprays to achieve special effects or properties. Included among these are lanolin derivatives, sunscreening agents, silicones, vitamins, fluorescent dyes, pigments, and soluble dyes. Lanolin derivatives sometimes are added just to substantiate advertising claims. Sunscreening agents in hair sprays have been found to have little value in protecting the hair from the harmful rays of the sun, mainly because so little of the hair is covered by the hair spray. Immersions of individual hair fibers in sunscreening agents is effective in protecting the strand from the damaging effect of ultraviolet light, but this effect cannot be demonstrated in actual usage of the hair spray with such additive. Hair luster after use of hair spray can be improved by the addition of silicone oils. These oils need not be compatible with the resin; but preferably plate out on top of the resin film, thereby imparting a luster to the hair. Panthenol and vitamins have been suggested as hair spray additives, but it has been difficult to demonstrate any effect.

Colored hair sprays have appeared on the market containing fluorescent dyes, pigments, and alcohol-soluble dyes. Such products have not met with much success in the United States, but have been popular in foreign countries, particularly Australia. The chief difficulty with this type of product is in its application. It is not possible to confine the spray only to the hair. The development of a practical method of application might make the colored hair spray products much more acceptable.

Solvent

Practically the only solvent for hair sprays has been anhydrous ethyl alcohol, specifically Specially Denatured Alcohol #40 (SDA #40) anhydrous. Very early in the development of aerosol hair sprays it was realized that 95% alcohol caused corrosion in metal containers, and therefore anhydrous alcohol was soon adopted as a solvent. The one drawback of ethyl alcohol is its flammability. By keeping the percentage of ethyl alcohol at 35% or lower in the aerosol hair preparation, it is possible to make the spray nonflammable according to Interstate Commerce Commission Specifications.

Other solvents which have been suggested and used with ethyl alcohol, which are non-flammable, are methylene chloride and methyl chloroform. Both these solvents, however, have an undesirable odor and are readily hydrolyzed by small amounts of water. This hydrolysis results in hydrochloric acid formation with consequent container corrosion and perfume deterioration. Trichlorofluoromethane (Propellent 11), although also susceptible to water hydrolysis, is more stable at low concentrations of water.

Propellents

All aerosol hair sprays contain a propellent. A mixture of Propellent 11 (trichlorofluoromethane) and Propellent 12 (dichlorodifluoromethane) ranging from 35% to 50% of the latter is generally used. The total amount of propellent ranges from 25% by weight of the formulation to 50%.

For aluminum containers, a mixture of Propellents 12 and 114 (tetrafluorodichloroethane) is recommended. Propellent 11 is particularly corrosive to aluminum.

The pressure in aerosol hair spray metal containers usually ranges from 20 lbs./sq. in. gage to 40 lbs./sq. in. gage at 70°F. When glass bottles are used, the pressure is kept below 25 lbs./sq. in. gage at 70°F.

Nitrogen, because of its low cost, has been suggested as a hair spray propellent; but it does not provide the flash evaporation afforded by the liquid propellents and hence the spray is wetter and the drying time is longer.

The hydrocarbon propellents have also been suggested as propellents for aerosol hair sprays. Their flammable and explosive character, however, mitigates against their use in personal products used in the home where, under some conditions, usage could be hazardous.

Perfume

The perfume used in aerosol hair sprays is particu-

larly important. The first, and perhaps the most important, reaction that a woman has to a hair spray is its fragrance. She is much more likely to be critical of the odor than the functional characteristics of the product. A good perfume will help to cover the odor of the solids in the hair spray, as well as the alcohol and propellent, and yet be mild enough so that it is not objectionable. Also, the fragrance should not stay very long on the hair where it may clash with the perfume used.

The trend in the past ten years has been towards the development of tailor-made resins that specifically meet the needs of a hair spray product. Whereas shellac and PVP may have been good enough in the first few years of the hair sprays development—the highly competitive market faced by hair sprays today is demanding new and better resins. What perhaps is the ultimate in hair spray products will be one that can be used on pin curls to provide a permanent wave and then used on the hair after it is styled to hold it in place. This is expecting a good deal from one product, but looking at the progress that has been made in the first ten years of aerosol hair sprays—anything is possible in the next ten.

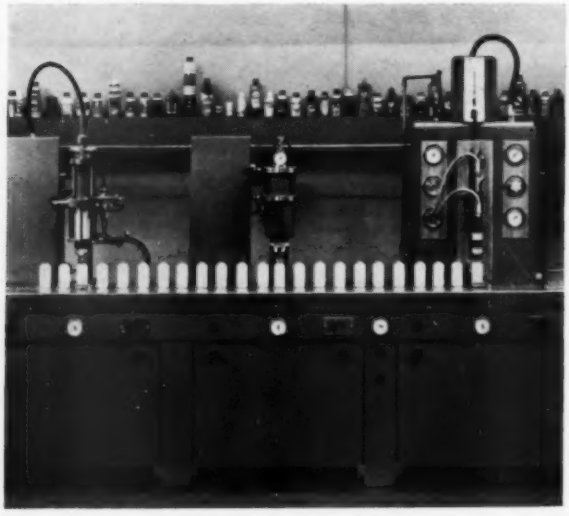
[1] U. S. Patent #2,871,161
[2] U. S. Patent #2,948,656

Vaniti-Mist is the name of a new fragrance aerosol container that features a reusable outer case, with a coated glass inner container. The inner container holds the fragrance and the aerosol valve, and may be replaced when it has been consumed.

The package above is Oh La La, by Parfums Ciro. The outer container, valve and actuator are by Risdon, the coated glass container is by Wheaton Plastics Co., and Aerosol Techniques, Inc., is the filler.

One-piece extruded aluminum aerosol containers by Peerless Tube Company have made a market for themselves in the smaller sizes of aerosols. The illustration above shows the unique construction features that have made it so popular with the cosmetic industry.

After a container has been extruded, trimmed and coated, its shoulder is formed and neck curled inward. It then appears in the form at left and as shown in the first cutaway. A cup-shaped top is then inserted with sealing compound so it interlocks with the curl around the container mouth, as in the second cutaway. The right hand cutaway shows how the dispensing valve is seated in the container mouth, sealed with a rubber gasket to further insure the container against leakage. End product, Yardley of London's "Flair" spray cologne, is at far right.



Druk-Pak-Waldherr aerosol line

Manufactured by Johann Waldherr OHG of Germany, and sold by Druk-Pak, Inc. of Switzerland, this line is the most widely used in Europe. It is all compressed air operated, and capacities from small manually operated to large entirely automatic lines are available. With a quick gassing valve, up to 40 units per minute can be filled on this single head machine. These units are available separately, as well as in complete lines.

Powder aerosols

By Victor DiGiacomo
Givaudan-Delawanna, Inc.

Keeping pace with the aerosol has not been easy. This cinderella of the packaging media has truly donned her glass slippers and has danced her way into the daily habits of almost every family in this hemisphere. The aerosol is as familiar a sight in the home today as was the old "flit" gun of years ago, but the aerosol is definitely here to stay. In this era of pushbutton living, the aerosol container has built quite a niche for itself . . . one that continues to grow each year. Today, the aerosol can be used for a multitude of functions . . . for insect control, the job that started the whole thing, to help curl the hair and to top off a freshly baked cake. The aerosol is even replacing the powder box and puff and milady can now spray on a fine mist of dusting powder when she leaves the bath.

Powder or "solid" aerosols first came into being when the pressurized paints and an aerosol fire extinguisher were developed. These showed the way for the possible application of the pressure package for proprietaries and personal products. The advantages of using the aerosol package were immediately obvious. Aerosol dispensing allowed for a better uniformity of the mix and prevented oxidation of the fragrant oils used as a perfuming medium. It became possible to retain sterility of a product for long periods of shelf life and also while the product was actually in use. Materials which were normally hygroscopic lost this tendency when they were aerosol packaged. Finally, pressurized powders could be easily applied in even, uniform layers on the area of application. Unfortunately, in spite of the desirability of the aerosol for powder products, not too many have reached the market when compared with the deluge of other products that have been pressurized, and those that are now available have been introduced in comparatively recent months. The reasons for the delay and hesitation in releasing powder aerosols were the several formulation problems that were experienced by all aerosol manufacturers in the very beginning. Those involving powders were, however, somewhat more involved.

As in all aerosols, the primary problem involved in adapting powders for pressure packaging was the reformulation which became necessary. It was essential that the particle size be such so as to readily allow dispersion through the valve orifice. In our own experimentation, we ascertained that a 325 mesh talc was most desirable for this purpose. Improperly formulated powders had a tendency to form agglomerative sedimentation, which we shall touch upon again, which prevented dispersion of the product from the container. It was also necessary to ascertain the compatibility of other materials which could be used in conjunction with the talc such as bacteriostatic agents (for deodorant powders), perfume constituents and others, with the suspending medium to insure proper valve function. Experimentation has made it possible to now prepare pressurized powder formulations which contain high concentrations of solids depending, of course, on the type of product to be prepared and the concentration of co-solvent utilized. The propellant mixture must be carefully selected to meet the needs of each individual product.

Although valves are important in all aerosol containers, they are even more important where powders are concerned. It is absolutely vital that the valve not be affected by the solvent which is used as the suspending medium. Today, such valves are available. The vapor trap valve, for example, is excellent, for this application. This valve allows the can to be used in any position and permits complete evacuation of the container.

The problem of agglomerative sedimentation, which we mentioned before, is one which can cause difficulties in a powder aerosol. It can, however, be prevented by the proper use of propellant mixtures and by selecting a suspending medium which can be mixed with the propellants and yet not affect the active principle of the product. The suspending medium must be soluble in any and all propellants that might possibly be utilized; must be odorless; be compatible with perfume ingredients; be greaseless and non-irritating; and must be usable in rather low concentrations. It must, of course, have no deleterious effect on the component

parts of the container and should allow the formulation to be readily dispersed on shaking without the use of a mechanical device.

By using a purified grade of isopropyl myristate, excellent results can be obtained. It becomes possible to suspend powders in aerosols and to control the type of spray. Concentrations ranging from 0.5 to 2.0% can be used depending on the type of product involved and the adhering properties desired. The high concentrations permit the spraying of material with a higher residual character permitting adherence of the product to the area of application.

As a result of the work done, it is now possible to package many different types of powder products in aerosols. Girdle and talcum powder aerosols are already a reality as are foot and pet powder sprays. Aerosol deodorant powders are a certainty, and we will no doubt soon see leg makeup and even facial makeup sold nationally in aerosol containers. A typical powder aerosol formulation is this deodorant spray.

0.05	G-11® (Hexachlorophene USP)
0.325	Deltyl® Extra (isopropyl myristate)
0.025	Perfume Oil
7.60	Talc USP 325 Mesh
92.000	Propellants 11 & 12 (65/35 mixture)
100.000	

In addition to the work already done on toiletries, a considerable amount of research has been performed in other fields. One example of this has been toward the use of starch in an aerosol. In some instances where the use of talc was not feasible, starch has been substituted with excellent results. This has been particularly helpful for pharmaceutical powders where starch is used to spray surgical rubber gloves and other rubber goods, particularly where sanitizing properties are desirable. Where starch is used, the basic formulation may be somewhat different from the talc spray. It is possible to reduce the concentration of the suspending medium without affecting the operation of the container.

Another area in which the aerosol seems to be catching on rather quickly and successfully is the shoe spray. This is somewhat similar to the basic powder aerosol except that the pigments are in a high liquid solvent medium. The product is evacuated as a wet spray for adherence to the shoes.

We should also mention aerosol sprays used for hair decorations and for theatrical makeup on both hair and body. The hair sprays permit the application of metallic particles on the hair to achieve the temporary effects desired.

Aerosol bath powder, one of the newer aerosol products, is being marketed by F. Millot in this Crown Spra-tainer. Aerosol powders are particularly appropriate for travel and beach use, as they eliminate the spilling and accidental opening that is often the case with the usual powder containers.

The hair care field, long the prime field of aerosol packages among cosmetics, received another boost by the introduction of a cold waving lotion and neutralizer set for the beauty salon market. If the convenience of this method proves to be of great importance to beauty operators, the whole market could swing to this method of packaging very quickly.

Chemical problems concerning the perfuming of aerosols

By Dr. P. H. Witjens

Polak & Schwarz International N.V.

When the first insecticide aerosols came into production about fifteen years ago, one certainly did not foresee that, in only a few years time, this type of packaging would develop into an important branch of industry covering a wide range of technical and household products, the fields of pharmacy and cosmetics and even food products. Neither would one have realized that such an interminable series of problems lay ahead, fundamental problems as well as problems of a more practical nature which have been worked on in the industrial laboratories and are still being studied.

The appearance of a book devoted entirely to pressurized packaging gives a clear idea of the extent of the problems that this way of packaging has brought with it.

Everyone who has followed the development of the aerosol industry closely would agree that the study of the more practical problems has had priority but only a very small part of the results of these studies has been published and this, although regrettable, is quite understandable. The research work mentioned in literature deals much more with the fundamental aspects of the aerosol field and it is striking that most of the studies of fundamental aspects deal with the physical properties of the propellents and the physical relations between the propellents and the other components of the aerosol. The chemical inertness of the propellents will certainly be one of the reasons for the emphasis being laid on the physical properties. However the rather slight chemical reactivity of the propellents does, on the other hand, play a big part in the problems concerning the aerosol in that it effects the stability of the pressurized product. I would like to illustrate a single aspect of this in greater detail to show you one of the ways in which a change of the aerosol and especially of the odour develop.

As a matter of fact, a change of odour, especially of the perfumes of cosmetic aerosols, can give the first indications that the contents of the container are not completely stable and generally precedes more serious chemical changes which can make the pressurized product completely unusable.

For this purpose, experiments were carried out using a system that could serve as a prototype for a special group of aerosols with a substantially cosmetic application, this system was based on:—

Propellents
Oxygen
Water
Ethyl Alcohol
Container Material

The propellent used for products of this group is usually a mixture of difluoro-dichloro-methane and monofluoro-trichloro-methane.

The most important purpose of the latter is to reduce the pressure of the mixture to such an extent that it falls below the value considered safe for commercially-available tin plate aluminium containers.

All the afore-mentioned components (Mixture 1 Table I) are brought into contact with each other, care being taken that the liquid phase is homogeneous. The mixture is then stored under two different sets of conditions, one at 100°C. for seven hours, the other at 55°C. for 14 days.

These experiments brought to light some interesting results which, furthermore, could explain the typical change of odour often detected in the first stages of aerosol corrosion and decomposition. Apart from a lowering of the pH of the mixture which could be expected under such conditions, the presence of acetaldehyde and acetaldehyde diethyl acetal could also be demonstrated. As the quantities of these reaction products formed were very small, their presence in the reaction mixtures was determined by gas chromatography analysis. This method as you know is based on the chromatographical separation of the mixture. The presence of the various components in the gas mixture emerging from the column being shown by changes in the heat conductivity of the gas stream which is continually being registered.

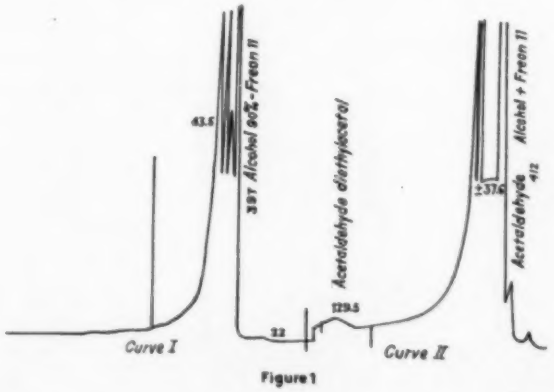


Figure 1

The untreated starting mixture No. 1 (See Table 1) gives curve 1, figure I on gas chromatography analysis. After heating the mixture for 7 hours at 100°C, curve 2, figure I was obtained. Two new peaks are clearly observable in this curve, one located before the peak for chloro-trifluoro-methane and the other, a broad peak, located after the maximum for ethyl alcohol. It could be proved that the former peak is caused by acetaldehyde by adding some acetaldehyde to the reaction mixture and obtaining a heightening of this maximum.

(Figure II) Acetaldehyde diethyl acetal was identified by locating the maximum with the pure compound.

(Figure III) Both these reaction products could also be unmistakably detected, though in much smaller quantities in Mixture 1 which had been stored at 55°C. for a fortnight.

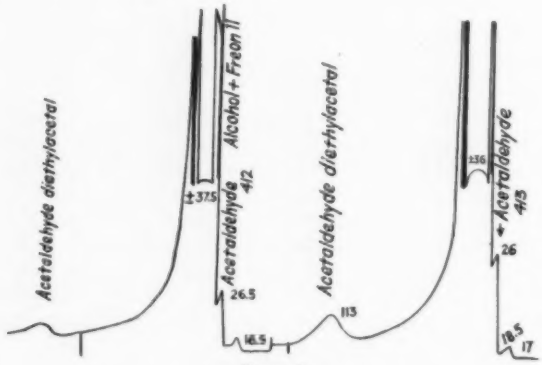


Figure 2

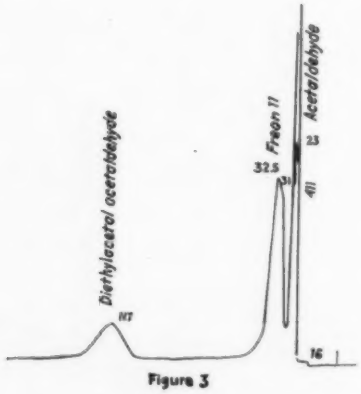


Figure 3

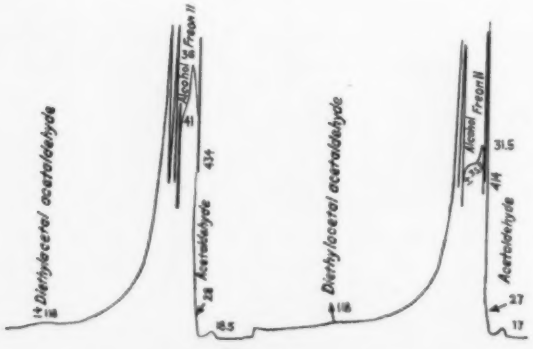


Figure 4

(Figure IV) The lowering of the pH, which has already been mentioned, is due to the formation of hydrochloric acid from the fluoro-trichloro-methane, and in both experiments a value between 3 and 4 was observed.

An attempt, based on these results, is being made to define the conditions under which these products are formed. From the observations mentioned the following hypothesis on the reaction-pattern can be deduced:

Under the influence of acid, formed from water and fluoro-trichloro-methane, and metal or metal halide, the ethyl alcohol is being dehydrogenated to acetaldehyde and this in turn is partially converted into the corresponding diacetal. The atmospheric oxygen can be primarily considered as the hydrogen acceptor.

This supposition is being tested in a series of experiments mentioned in the next table.

Table 1

	$CFCl_3$	H_2O	EtOH	O_2	temp. °C.	pH	acetaldehyde
Mixture 1	50	5	45	+	55°	336h 2	++
Mixture 2	50	5	45	—	55°	336h 4-5	+
Mixture 3	50	5	45	+	100°	7h 3	++
Mixture 4	50	0.05	50	—	100°	7h 4-5	—
Mixture 5	50	5	45	—	100°	7h 3-4	+

Acetaldehyde : detectable by gas chromatography ++
Acetaldehyde : only detectable by odour +
Acetaldehyde : not detectable by odour —

These experiments, all carried out in the presence of tin plate, show clearly that atmospheric oxygen promotes the formation of acid as well as acetaldehyde but its absence does not however exclude the processes completely. The substantially complete absence of moisture depresses the formation of aldehyde almost entirely although some acid is still being formed.

In aerosols containing alcohol and the propellents 11 and 12, the above mentioned processes may also be present in practice. However the process of decomposition does not stop here. The aldehyde formed will then condense, under the influence of an acid-metal catalyst, to form unsaturated condensation products which together give the typical corrosion odour which these aerosols can possess.

If the products in question are perfumed, thus containing a complex of aromatic chemicals as a part of the active ingredient, it is clear without further explanation that a lot of other condensation, esterification, re-esterification and acetalisation reactions can take place, by which the character of the perfume composition can be radically changed.

All these processes can take place before corrosion actually occurs and before the aerosol is unsaleable for that reason. Qualitatively however the product, especially when it is destined for cosmetic use, is already inferior.

It can be concluded that in the formulation and filling of monofluoro-trichloro-methane containing aerosols, the presence of products which can act as a hydrogen acceptor must be excluded as much as possible. As far as pressure filling is concerned this means that it is recommended that the air present in the container is removed or replaced by an inert gas.

Methods for dispensing aerosol flavoring preparation

By Philip Meshberg
Emson Research, Inc.

The number of flavoring preparations adaptable to packaging in aerosols, or more accurately, under pressure, covers an extremely wide range. It includes seasonings such as garlic and others, food concentrates, synthetic flavorings, coffee, tea and many others, as well as mouth rinses (antiseptic) and breath fresheners.

The potential in this pressure packaging field is limitless, and the advantages many. The chief advantage relates to the fact that most food products deteriorate in fragrance, taste and potency due to exposure to air. Once opened, the conventional package becomes subject to certain conditions which can, in time, cause spoilage.

In contrast with this, the product packaged under pressure can be dispensed as needed over a long period of time. The remainder of the packaged product remains un-exposed to air, thus retains its freshness and flavor for extended periods of time. It can be used days, weeks or months after it was first opened and often does not have to be refrigerated to prevent spoiling. Thus, with pressure packaging it is possible to have fresh coffee at your fingertips at all times, without having to worry that the similarly packaged cream you add to it may have become sour.

Further, exposure to air and certain temperatures tends to alter the physical appearance of many foods—ketchup, salad dressings, preserves and even that mealtime mainstay, butter. It is possible that pressurized packaging can maintain these products in a manner that will permit their use in their most appetizing form at all times.

Considering these factors and their favorable impression upon the consumer, one can appreciate the impact that pressure packaging of foods and other flavorings is making upon the manufacturers of such products. Their interest is even greater in view of the fact that progress made thus far in the pressure packaging field indicates that anything that can be scooped out, poured out or squeezed out, can probably be propelled out of an airtight container through the medium of a valve in an economical manner and in a controlled fashion offering innumerable consumer advantages.

But there is still much work to be done before this limitless potential can be realized. Adapting of flavorings to a pressurized package is a long and laborious process in which each product, each type of flavoring, must be subjected to rigorous tests and trials. To date, no one pressure package—container, propellent and valve assembly has been perfected to work with all flavorings.

One key factor governing development of pressure packages for flavorings is the choice of propellent. Liquid propellents such as Freon, Genetron, Ucon and Isotron raise an element of toxicity which, at this writing, has not been overcome in obtaining full approval for use of these propellents in direct contact with food products. Compressed gases such as CO_2, nitrogen and nitrous oxide have been used with certain food products, the more common being whipped cream, desert topping, toothpaste, mouth rinses and certain others.

A new package for dispensing a controlled amount of flavoring has been developed by this writer for use with compressed gases (see figure 4). Its advantages

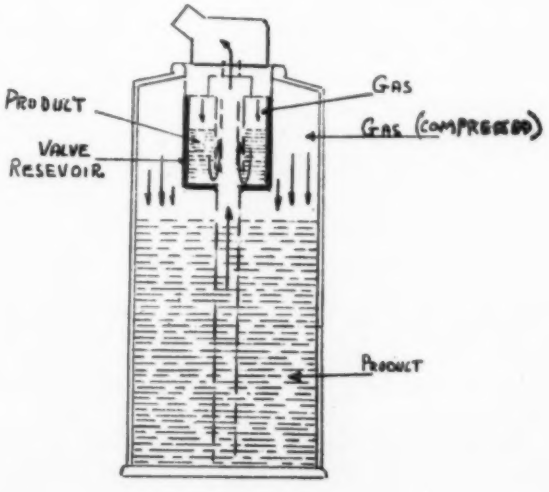


Figure 1

Figure 2

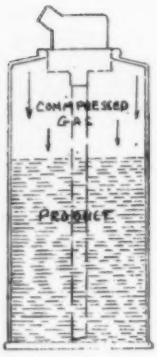


Figure 3

are not only the ability to dispense a controlled amount of the product, but also to dispense the entire contents of the container. Also, there is no possibility of accidental discharge of gas along with the product if the package is held improperly when dispensing. These are all common failings with many of the present flavoring dispensers that use compressed gases (see figure 3).

The newly developed metered dispenser has a large reservoir as part of its valve assembly. The compressed gas at the top of the container pushes the product through the dip tube into the valve reservoir which, in turn, builds up a dispensing pressure. When the valve actuator is depressed, the dip tube is closed off from the body of the container and the material trapped in the valve reservoir under pressure is evacuated through the actuator. When the valve is released, the inlet port leading from the dip tube is again opened and the valve reservoir is refilled for the next operation. In principle, when the valve is opened, the product is dispensed from the valve reservoir and not from the container. Therefore, when the valve is opened, it is impossible to accidentally discharge the gas, regardless in which position the container is held.

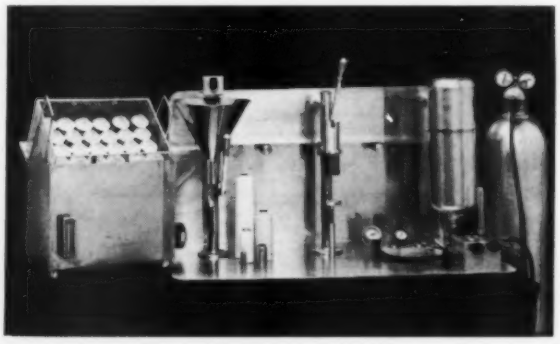
This process of refilling the valve reservoir after each application assures the siphoning up of all of the product remaining in the container. Thus, all of the product which can possibly be evacuated is siphoned out. The valve reservoir size is pre-determined by the amount of the product which is required per application to dispense the product properly.

Until such time as approval is granted permitting use of liquid propellent gases in direct contact with flavorings, dispensers using these gases must have an artificial barrier between the propellent and the product. At least one such container has been tested. In figure 1, just such a device is depicted. A movable piston which clings to the walls of the container, pushes against the product. Pressure is created by the trapped propellent, located at the bottom of the container. As soon as the valve is opened, the product is forced out through the dispensing head.

Another dispenser which uses a liquid propellent and which isolates it from the product is depicted in figure 2. An expanding bag made of rubber or plastic is filled with the product. Gas is introduced into the container surrounding the bag and exerts pressure on the outside surface of the bag. When the valve is opened, the pressure of the surrounding gas forces the product through the valve opening and out through the dispensing head.

These are some of the possibilities that can be explored for pressure packaging of flavorings. But other key problems exist. In using inert and compressed gases at pressures of 100 pounds or more, selection of the proper container and valve are important. Since such pressures are frequently employed, a variety of containers are now in use and others are being tested. In addition to the conventional drawn type, there are one and two-piece aluminum containers which can be purchased in sizes from ½-oz. to one quart. The manufacturers of glass plastic-coated bottles are experimenting with larger capacities with openings for faster filling.

The potential offered by pressure packaging of flavorings is so great that we shall continue to see extensive developments in all phases in a continuous effort to achieve dispensers that will offer the consumer practical, attractive and reliable packages that will maintain and improve present standards in packaging and dispensing the wide range of flavorings that include foods, mouth washes and fresheners, perfumes, colognes and other products where a fragrance or aroma is involved.



By Pressure Pak, Incorporated

A complete pressure filling unit has been developed, including automatically controlled filler, crimper for both cans and bottles ranging from one ounce to eighty ounces, and a combination fluorocarbon and nitrogen gasser. Drip, foam, powder, stream, spray, mist and metered valves are compatible with this unit.

Manually operated, the unit is intended for experimental and laboratory, as well as very short run use. It accommodates all pressure-fillable products, needs little space (only eight square feet), and requires no specialized technical knowledge to operate. This basic unit sells for about $400, with supplemental equipment, such as pressure vacuum testers, lead test equipment, propellent racks, etc., available at correspondingly low cost.

Inhibition of metallic corrosion in aerosols

By K. Klausner
General Chemical Division
Allied Chemical Corporation

The problems of metallic corrosion in cosmetic aerosols cannot be overemphasized. Since the aerosol system must contain, and be able to dispense the product for its expected commercial life, the corrosion resistance properties of the container, along with the product's stability, must receive the most thorough technical analysis before the product is released for marketing. Papers by Goodhue and Young on the stability of aerosol formulations have shown that certain additives can inhibit metallic corrosion in an aerosol system (1, 2).

This paper is essentially a preliminary review since it deals primarily with the theories behind the use of corrosion inhibitors. As information from the stability tests now under way at General Chemical's laboratories is obtained, it should show how the use of specific inhibitors in different types of aerosol systems can render them less corrosive to metallic containers.

Metallic corrosion

To understand the theories behind the use of certain corrosion inhibitors, one should first understand how and why metallic corrosion occurs. An understanding of corrosion is provided by the following simplified, short review.

It has been firmly established that metallic corrosion in an electrically conducting liquid media is of an electrochemical nature (3). The reactions that occur are governed by the same chemical laws that apply to electroplating, or the functioning of batteries. In a corrosive environment, one finds countless local galvanic couples forming on the surface of the corroding metal; the metal is spontaneously oxidized and some substance in the surrounding environment is reduced. The basic requirements for such reactions are always (4):

1. Differences in potential between adjacent areas on an exposed metallic surface to provide anodes and cathodes.
2. The presence of moisture to provide an electrolyte.
3. A corroding agent to be reduced at the cathode.
4. An electrical path in the metal for electron flow from anodes to cathodes.

At the anodes, metal is oxidized and metallic ions enter into solution. At the cathode, the corroding agent is reduced (here we have the consumption of electrons). Using iron as an example, the reactions that take place are:

At the anode

1. $Fe = Fe^{++} + 2e$

As the metal ions move away from the surface, they encounter hydroxyl ions in the surrounding electrolyte and form precipitates of corrosion products. Therefore, the overall anodic reaction is:

2. $Fe + 2OH^- = Fe(OH)_2 + 2e$

Depending on environmental conditions, the precipitate is gradually oxidized to ferric hydroxide and eventually dehydrated to the iron oxides commonly found in rust.

At the cathode

Here several reactions are possible depending on the environment. In the absence of air, hydrogen-type corrosion occurs (hydrogen gas is evolved). In an acid media, Reaction (3) predominates:

3. $2H^+ + 2e = H_2$ (gas)
4. $2H_2O + 2e = H_2$ (gas) $+ 2OH^-$

When air is present, oxidation (with the corresponding reduction of oxygen) is more spontaneous than hydrogen evolution. In oxygen-type corrosion, under acid conditions, Reaction (5) predominates; in neutral or alkaline environments, Reaction (6) is dominant:

5. $O_2 + 4H^+ + 4e = 2H_2O$
6. $O_2 + 2H_2O + 4e = 4OH^-$
7. $O_2 = 2H_2O + 2e = H_2O_2 + 2OH^-$

Reaction (7) does not occur to any appreciable extent and is presented only as a point of information.

Polarization

As pointed out before, corrosion of metals in the presence of moisture takes place through the dissolution of the metal at the anodic areas. The electrons

This paper was presented at the October 13th, 1959 meeting of the Chicago Chapter of The Society of Cosmetic Chemists.

released at the anodes are transferred to the cathode through metallic conduction and are consumed there by the cations present in the solution. In this way, a current flow occurs through what can be called the corrosion cell. However, when current carried by ions flows to or from a metal surface, certain irreversible effects generally occur adjacent to the metal surface (3). These irreversible effects always oppose the direction of current flow. For convenience, all these irreversible effects can be grouped together and called "polarization." In a battery, where the objective is the efficient conversion of chemical energy to electrical energy, it is important to minimize polarization. However, in electrochemical corrosion, a high degree of polarization is desirable since it slows down the rate of attack (5). This occurs because the corrosion rate is directly related to cell current per unit area. Therefore, any reduction of cell current will tend to inhibit corrosion.

The equation for corrosion current (following the treatment by Mears (6)) is:

$$I = \frac{E_o - (E_a + E_c)}{R}$$

I = Corrosion current
E_o = Open circuit potential difference between local anodes and cathodes.
E_a = Anodic polarization
E_c = Cathodic polarization
R = Resistance of the electrolyte between anode and cathode areas.

To reduce corrosion current, the open circuit potential (Eo) can be decreased or the anodic polarization (Ea), cathodic polarization (Ec), and/or resistance (R) can be increased. Eo can be reduced by rendering the surface more homogeneous (by polishing, etching, using a purer metal, etc.). However, the usual approach to inhibition is to add substances which will increase Ea or Ec or both.

Type of inhibitors

Corrosion inhibitors inducing anodic polarization (by retarding the anodic reactions) are classified as "anodic inhibitors," and corrosion inhibitors inducing cathodic polarization (by retarding the cathodic reactions) are classified as "cathodic inhibitors." There are also a number of substances which inhibit metallic corrosion by interfering with both anodic and cathodic reactions, thereby causing both anodic and cathodic polarization—or mixed polarization. Gelatin, glue and other high-molecular-weight organic compounds are such substances and are classified as "general inhibitors" (7).

Cell potential relationships are shown in Fig. 1 and Fig. 2 in a rather simplified form. It is assumed here that the electrolyte resistance is small and that electrode potentials vary linearly with current.

In Cell 1, an ideal case requiring an adequate oxygen concentration or where hydrogen is readily discharged, there is no appreciable polarization of either anodic or cathodic areas (as indicated by the small change in potential); the corrosion current flow is limited only by the resistance of the electrolytic paths between anodes and cathodes. However, in most corrosion cells, after a period of time, the potential of the anode or the cathode will shift as the particular electrode becomes polarized. The anodic potential shifts toward more cathodic values, and the cathodic toward more anodic values.

In Cell 2 there is both anodic and cathodic polarization, or mixed polarization. The addition to this cell of either (or both) an anodic or cathodic inhibitor which induces further polarization will reduce the corrosion current equally. However, each particular inhibitor shifts the potential in the opposite direction.

Cell 3 is under anodic control while Cell 4 is under cathodic control. To verify that Cell 3 is actually under anodic control, it is noted that in the presence of an anodic inhibitor the resulting potential of the cell moves in a cathodic (or more noble) direction as the process of inhibition takes place; it should be kept in mind that shifting of the potential in Cell 3 to more cathodic values upon addition of an anodic inhibitor is indicative of anodic polarization if accompanied by a decrease of the corrosion rate.

However, it should also be kept in mind that reduction of the total cell current is not the main criterion in choosing the proper inhibitor. One must also consider the effect on current density (8). Suppose in a system under cathodic control (Cell 4), an anodic inhibitor is used thus reducing the anode area, but reducing the cell current only slightly. Since the controlling factor in this cell is the cathodic reaction, the same amount of corrosion must now be distributed over a smaller anodic area, resulting in intensified localized attack (or pitting). It is therefore apparent that if the anodic inhibitor is *not* added in sufficient amounts to maintain complete passivity over the entire anode area, accelerated corrosion can actually occur since the current density at the exposed anode areas is increased. Thus, when improperly used, anodic inhibitors can be dangerous.

On the other hand, cathodic inhibitors, with a few exceptions, do not lead to intensified or localized attack. This is because the cathode areas are not attacked during corrosion, since the metal itself does not participate chemically in the cathodic reactions. If corrosion is under cathodic control, a decrease in the cathode area due to partial inhibition will result in an over-all decrease in corrosion. If the cell is under anodic control, a decrease in the cathode area will increase the cathode current density, but will have no effect on the over-all corrosion or its nature. Because of this, cathodic inhibitors are usually referred to as safe inhibitors since they exhibit no adverse effects on the corrosion system, even if employed in insufficient amounts.

Potential measurements

It should be noted that the observed potentials of corroding metals represents the average (or composite) potential of the local anodes and cathodes on the metal surface; the value of this potential is determined by the intersection of the anodic and cathodic polarization curves (the convergence points).

It is apparent that a knowledge of the anodic and cathodic current density–potential relationships would make possible an understanding of the processes which are taking place and lead to a prediction of

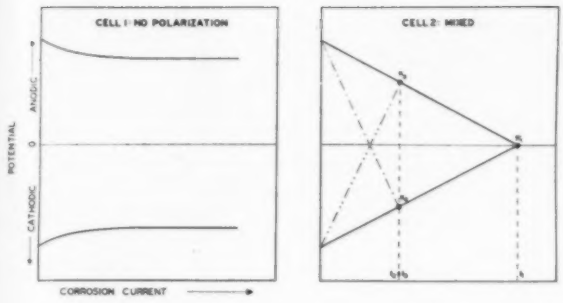


Figure 1

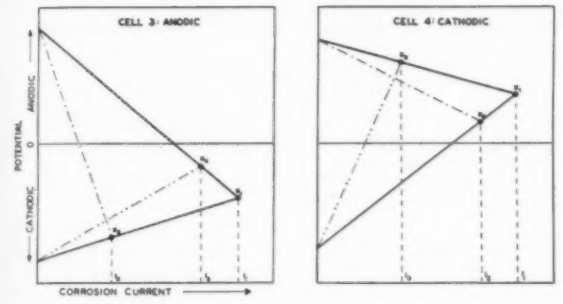


Figure 2

corrosion behavior (9). However, only by determining a composite of the resultant potentials and their change with time (using a recording potentiometer) can this be done.

With aerosols, it has been found to be extremely difficult to obtain accurate potential measurements of the complete system. The usual method is to determine the electrochemical potential between metallic components of the aerosol container and the product itself. But even here, the addition of a perfume or a resin can alter the readings drastically. Also, there is no indication of what is occurring in the vapor phase. In most cases, it has been necessary to rely on storage tests and visual and microscopic examination of the corroded surfaces.

Classification of inhibitors

In classifying the corrosion inhibitors, it is assumed that each type does not interfere with any of the other reactions. Although such assumptions are often valid, it should be kept in mind that many anodic inhibitors, to some extent, cause cathodic polarization and vice versa. Such inhibitors are still considered as anodic or cathodic depending upon the electrode at which their action is more pronounced (7).

1. Inorganic inhibitors

A. Anodic inhibitors

Various mechanisms have been proposed to explain the action of inhibitors. Some controversy still exists particularly with respect to inorganic anodic inhibitors. One view is that they form protective insoluble films on metal surfaces (10). Another states that they merely adsorb without forming definite compounds (11). Strong evidence for the oxide film theory derives from tests with nitrites, chromates, phosphates, and molybdates in aerated solutions. However, measurements of potential in the presence of chromates, molybdates and tungstates support the adsorption mechanism.

In an aerosol system, the exclusion of air would tend to inactivate those anodic inhibitors that did not contain oxidizing anions (following the oxide film theory). Therefore, it is assumed that the only anodic inhibitors that would possibly work in a completely air-free aerosol would be nitrite, chromate, and to some extent, molybdate and tungstate.

Inorganic anodic inhibitors include the following: Borates, benzoates, carbonates, chromates, dichromates, molybdates, nitrites, permanganates, phosphates (hexa, meta, ortho, etc.), silicates, and tungstates.

B Cathodic inhibitors

In neutral solutions, salts of zinc, nickel, manganese and chromium are considered cathodic inhibitors for iron. These salts function by forming insoluble hydroxides which impede hydrogen ion reduction and oxidation reactions at the cathode areas (see cathodic reactions).

In acid solutions, the salts of arsenic, antimony and mercury act as cathodic inhibitors. It is believed that in this reaction metallic antimony or arsenic is deposited on the cathodic areas and decreases the hydrogen evolution by causing an increase in the hydrogen overpotential.

Because of the possibility of the formation of insoluble (and possibly toxic) products, the use of inorganic cathodic inhibitors should be very carefully screened before being used in an aerosol system.

Inorganic cathodic inhibitors include salts of the following metals: Antimony, arsenic, chromium, manganese, mercury, nickel, and zinc.

2. Organic inhibitors

Organic inhibitors, in general, do not undergo chemical changes during inhibition.

A. Polar organic inhibitors

These substances usually function by forming a protective film (due to adsorption) or by increasing the hydrogen overvoltage. Organic compounds containing nitrogen (amines, pyridines, quinolines, etc.), oxygen (aldehydes, ketones, acids) and sulfur (mercaptans, etc.) fall into this class. Quinoline and thiourea compounds have been found to inhibit either cathodically or anodically, depending upon the concentration in which they are used. In particular, the polar organic inhibitors have appeared to be the best type of inhibitor for use in aerosols.

Polar organic inhibitors include the following: Acids (oleic, stearic, succinic, etc.), aldehydes (formaldehyde, etc.), amines (morpholine, etc.), bone oil (Dippel's oil), ester or ether alcohols (hexynol, etc.), ketones, mercaptans (and other sulfur compounds), pyridines, quaternary ammonium salts, quinolines, thiourea.

B. High-molecular-weight organic inhibitors

These compounds function as general inhibitors to both anodic and cathodic areas. They form a porous layer on the metal surface, thereby increasing corrosion cell resistance. Gelatin, egg albumin, dextrine,

agar, glue and other high-molecular-weight organic substances of plant and animal origin have been used as inhibitors in both acid and neutral media.

Summary

In approaching the solution of an aerosol corrosion problem by means of an inhibitor, certain fundamentals should be noted.

1. A literature search for reports on the use of inhibitors in identical or similar systems is usually the first step.
2. If possible, one should attempt to find out what type of galvanic control the test cell is under. If feasible, potential measurements, using known anodic, and/or cathodic inhibitors, should be made, and the potential shift and current drop (if any) noted.
3. Enough storage samples for each variable plus an adequate number of control samples should be prepared.
4. In selecting an inhibitor, the general criteria should be:
   A. Whenever possible use the proper type inhibitor for the corrosion cell being investigated (i.e., for a cell under cathodic control use a cathodic inhibitor),
   B. the inhibitor should be compatible with the product being tested,
   C. the inhibitor should successfully perform its specific function over the service life of the product under the normal conditions of use.

It is our opinion that the general principles we have discussed should provide a sound background on corrosion and corrosion inhibitors, and that any corrosion inhibition testing program should be based on these principles.

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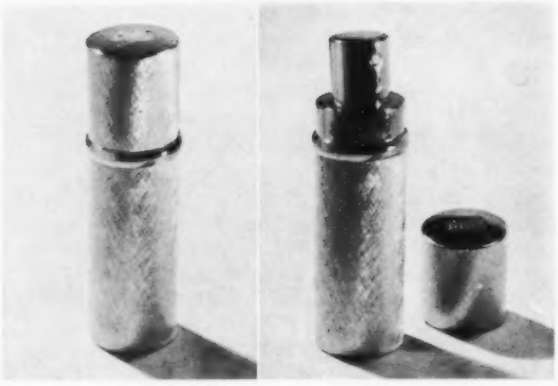
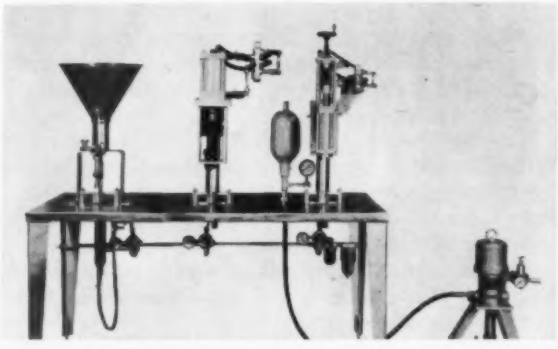
Purse size aerosols, called "Touch-N-Spray" by Richford Corporation, feature two types of containers. Style #1 provides a wide assortment of color and color combinations in refillable plastic containers. Two styles of spray heads with brass shell in polished or design background. Style #2 is called Aero-mesh, and features a basket weave plastic protector that adds a unique decorative note.



This reusable fragrance container is no larger than a lipstick. The metal outer container is available in a wide variety of finishes, and may be refilled by the consumer any number of times.

The coated glass inner containers, which hold the fragrance and valve, may be of one ounce or two ounce size. The Mini-Mist, shown above, contains Parfum Mist by Ciro, in six scents. Container and valve are by The Risdon Manufacturing Company.



Kartridg Pak Company

All types of aerosol packing lines, from low-cost semi-automatic to very high speed completely automatic, continuous motion, are available from Kartridg Pak. The newest item, the KP-1000 line is a completely self-contained unit, for producing up to 600 cans per hour. It is of the low-cost, semi-automatic type, yet is entirely practical for regular low volume production runs. It is composed of a piston filler, a vacuum crimper, and a pressure filler (0 to 300 grams). This line, or its individual units, are available for either sale or rental.

Aerosol containers

By Jack Pickthall, F.R.I.C.,
Polak & Schwarz (England) Ltd.

In many cases the choice of the container for a given aerosol product presents little problem. For instance, few people would quarrel with the selection of glass for a high-class handkerchief perfume, although the possibility of stainless steel should not be overlooked. If price considerations are of paramount importance and the product itself is suitable then blackplate might be an obvious choice. Unfortunately, the cheapest container may not be suitable for the product under review. Questions of corrosion, effects of product on container and container on product require careful consideration.

Without knowledge of the exact formula involved, it is impossible to classify specific types of products under their most appropriate container heading. For this reason it is advisable to discuss each type of container separately and to indicate some of the problems which may arise with various formulations.

Glass

If price and safety were not a consideration then glass might well be considered as the ideal container for pressure-packed products. The adverse effects of metal on an active ingredient and the possible corrosive effect of the constituents on a metal container are well known. The public, especially the ladies, appreciate glass as the traditional container for their perfumes and toilet waters and one cannot visualize them readily accepting a change to metal. This holds good at any rate for the presentation or dressing-table style of perfume container although the picture may be different where hand-bag sizes are concerned. For many pharmaceutical products it would be unwise to consider a container other than glass and it is chiefly in the medicinal field that expansion of the pressure-packing technique can be foreseen.

It would appear that glass is the "ideal" in aerosol containers, limited only by considerations of price, size and safety. When dealing with price this may be less important in the field of relatively expensive perfumes and pharmaceuticals. It would be unrealistic to consider glass containers of a size greater than 4 ozs. The question of safety has been discussed in the literature and there is now in most countries agreement on the need for an adequate plastic-coating on all glass aerosol containers. Plastic coated bottles may be obtained in a variety of attractive sizes, shapes and colours. Two interesting articles dealing with plastic coatings should be consulted the first by Nigel Vinson (1) and the second by A. R. Marks (2). Vinson claims that polyvinyl chloride is the only really satisfactory material developed by the end of 1958. He suggests that the plastic envelope should be at least 0.015 inches thick (other writers have suggested a minimum thickness of 0.025 inches). Its function is twofold: in the first place, the coating reduces the possibility of the bottle breaking when dropped. Secondly, if breaking does occur, then the plastic will expand to hold the rapidly expanding gas and then gradually collapse as the gas escapes through the small perforations in the coating. Vinson indicates that height control in the dipping stage of the plastic is all important as success is dependant upon the formation of plastic at the lip of the neck of the bottle. Other requirements are, a straight neck of at least 5 mm. between shoulder and top; a minimum 2 mm. punt in the base to allow surplus plastic to level off; edges should have a minimum ⅛th inch radius to prevent the coating thinning at these points.

A tentative method of "Drop Testing of Glass Bottle Aerosols" has been prepared by the Project Committee on a Standard Drop Test of Glass Aerosol, Scientific Committee, Aerosol Division, Chemical Specialities Manufacturers Association (3).

Although doubt was cast on the acceptability of high-class perfumes in containers other than metal this does not necessarily apply to the small hand-bag size. In fact, glass, stainless steel and aluminium are being used with some success. There is no doubt that exceedingly attractive and efficient packs are being offered to the public and they should find a very ready acceptance. The fact that the enclosed perfumes can be dispensed in measured doses is an added attraction.

Metal

For many years metal containers had the market to themselves and there is no doubt that they will continue to hold the major position. On the score of cost and safety they offer advantages over glass but perhaps they score chiefly on the ground that relatively large containers are possible. In appearance they are restricted by the fact that the shape must be basically cylindrical and of course in many

cases it is inadvisable to allow a product to come into contact with metal. Although the use of internal lacquering has done much to reduce adverse effects by metal on the constituents of a pack or adverse effects of the constituents on the metal, it is far from a complete answer. The lacquer itself may be attacked by the constituents and often corrosive attack will concentrate at pinholes in a lacquer and perforation will be even more rapid than would have been the case with an unlacquered container. Corrosion may be accentuated under acidic conditions by the presence of air in the system. In some cases it is advisable to double lacquer and even to reinforce by a final spray lacquering.

Blackplate

Blackplate containers (untinned steel) are used chiefly in the United States and Germany. Although some blackplate containers are made from sheets and possess a side-seam, the majority are made by drawing and are seamless. In the main, such containers are internally lacquered.

Tinplate

Tinplate consists of mild steel sheet covered with a thin coating of tin. Although extruded tin-plate containers are obtainable in some countries this is not so in Great Britain. Containers made from flat sheet possess a rather unsightly side-seam which does not lend itself to elegance in presentation. Unless a producer is prepared to use a paper label, the presence of a one inch wide strip bereft of decoration must be accepted.

It has been stated (4) that internally plain tinplate containers containing alcohol and trichlorofluoromethane have a shelf-life of 12-15 months at 77°F, providing the moisture content of the alcoholic solution does not exceed 1.5%.

Stainless Steel

It is possible to consider stainless-steel as a container for certain packs where price may not be too important.

Aluminium

The aluminium container monopolized the British market until 1955 when tin-plate containers became available. In Italy aluminium dispensers are still the most important of all aerosol containers. They are offered in many shapes and sizes and may be bulged, fluted, ribbed or formed into a variety of designs. Although mainly constructed with a dished double-seamed tin-plate bottom they are also available in "one piece" style. Aluminium offers several important advantages over tin-plate. It possesses a high degree of strength and formability and is very suitable to processes of lithographing and anodizing and as previously stated, may be processed in one piece. There are, however, certain disadvantages which must be taken into consideration and of these disadvantages corrosion is certainly the most important. In many ways aluminium containers are ideal for the packing of cosmetic products but unfortunately many cosmetic products will attack this metal. Thus ethyl, n. propyl and n. butyl alcohol should not be packed in internally plain aluminium containers. However, under these conditions iso propyl alcohol is safe providing the total moisture in the container does not exceed 0.05%. This holds good even when trichlorofluoromethane is present.

It is possible that some of the main constituents of shaving creams and shampoos will corrode aluminium although it has been stated that sodium lauryl sulphate in water has no ill effects in internally plain aluminium containers even in the presence of trichlorofluoromethane.

The internal lacquering of aluminium containers is more difficult than is the case with the tin-plate container. The lacquering of the latter is carried out on a flat plate but that of the former must be undertaken by spraying or flushing of the finished container, naturally, a more expensive process.

It is absolutely essential with aluminium to avoid pin holes in the lacquer film and in some cases, as many as four coats are necessary to overcome this occurrence. In the event of pin hole formation intensive corrosion will centre at any pin hole in the lacquer and perforation of the metal is much more likely than if the attack had been spread over a wide area of metal.

In Switzerland, the metal is protected by an anodising process in which a highly resistant oxide layer is involved.

DuPont (5) has shown that corrosion by anhydrous ethyl alcohol is more severe than by 99% ethyl alcohol although even in the latter case, corrosion can be severe. Two excellent articles on aluminium aerosol containers have been written by Aksel Taranger and these are worth consulting (6) (7).

Plastic

There is no doubt that a really satisfactory plastic container would solve a lot of the industry's present problems. First reference to a plastic container appears to be Cooksley's patent (8) although the nature of the plastic is not given. In 1955 containers made of melamine appeared in the U. S. The dispenser was moulded from melamine and contained a polythene dip-tube, a melamine valve housing a nylon valve stem and a polythene actuator. The sealing ring was made of buna rubber and a stainless steel spring in the valve was the only metallic part. In a report on a cologne packed in melamine (9) it was said to have "satisfactorily passed tests for breakability, product distortion and shelf-life". A warning was given that, "this plastic aerosol package must be designed properly and is reportedly safe with reasonably careful handling in use". On the question of permeability it was said that, "in six months study under elevated temperatures and other appropriate test conditions, samples showed no discernible loss".

Polythene was another material under consideration but serious doubts concerning permeability existed, even with high density grades. It was said that even the halogenated propellants permeated the plastic container. Progress was made when the nylon containers were referred to in 1956 (10). Further articles describing these containers appeared in 1957 (11) (12). In a later review (13) impressive performance details were given. To quote, "an analysis of certain factors would seem to indicate that the

Product	1st. Choice	Possible
Handkerchief perfumes	H	G, I
Toilet waters	H	I
Perfumes, handbag size	H, G, F	
Personal deodorants	C, F, H	I
Perfume foams	C, F, H	
Hand cleaners	B, D, E	
Suntans—based on Oil	A, B, C, D, E, F	
based on Alcohol	B, C, F	
Shampoo	B, E, F	
Shave-creams	D, F	
Hair lacquers	C, F	
Toothpastes	A, B, E, F	
Room deodorants	D, F	C, E
Anti-biotics	H	E
Burn-dressings	D, F	
Cold cures	E, F	
Moth proofers	A, C, E	
Anti-mists	D, F	C, E
Anti-statics	D, F	C, E
Polishes—spray	C, E	
foam	C, D	
Paints	C, E	
Paint removers	B	
Oven cleaners	D	
Body powders	C, E	
Nail polish driers	C	
Window cleaners	C, D	
Waterproofers	C, D	
Insect repellants	C, D, E	
Penetrating oils	A, C, E	
Spot removers	A, C, E	

Key:—

A Blackplate—internally plain
B Blackplate—internally lacquered
C Tinplate—internally plain
D Tinplate—internally lacquered
E Aluminium—internally plain
F Aluminium—internally lacquered
G Stainless steel
H Glass—plastic coated
I Plastic.

nylon container—the first all plastic container to reach commercial importance in the U. S. aerosol industry —is ready to become a major factor in pressure packaging of cosmetics". Nylon is light, inert and lacks potential breaking hazards. Costs may be relatively high but "the relative strength and mouldability of the material allowing manufacture of containers with wall thickness of 0.025 inches", offsets this to some extent. In the article quoted above (13) impressive experiments on the permeability of a cologne, a shampoo, two hair lacquers and two anti-perspirants are given. Also covered are permeation tests on propellants. Despite the satisfactory results the makers say "test each candidate formulation for a minimum period of three months in order to establish not only whether weight changes due to permeation are acceptable but also whether any adverse changes occur in the formulation".

The latest development in plastic containers is mentioned in an article written in April 1959 (14). The product "Delrin" is made from formaldehyde into what is called "a linear acetal resin". It is translucent but can be pigmented or decorated. It is said to offer the following advantages over many conventional packages: high strength styling, freedom from corrosion and light weight. The injection-moulded bottles possess high gloss. "Delrin" is said to resist all organic solvents, including the commonly used propellants and most aqueous solutions. The results of many stability and permeability tests are given in the article.

In considering plastic materials as aerosol containers the stringent rule which applies to all containers should be enforced. Take no chances but ensure that shelf tests are of sufficient duration and that they prove the complete efficiency of the *whole* pack. This means that proof must be forthcoming that both contents and container are unaffected.

Below is an attempt to indicate in the form of a chart the most appropriate container for a selection of aerosol products. To be definite in indicating a choice is impossible unless reference is made to the formula involved. A change in propellant, an increase in moisture, a change in pH etc., may completely change the picture.

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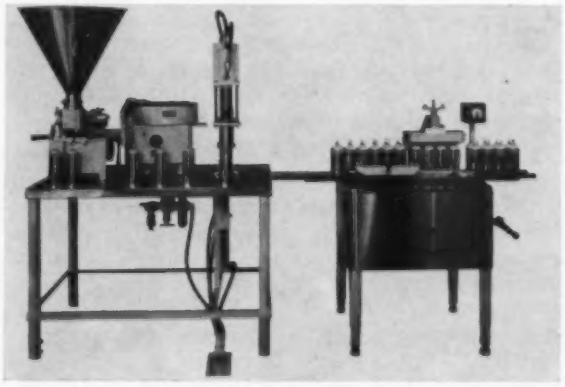

John R. Nalbach Engineering Company

This firm, essentially consulting engineers, was asked to design aerosol equipment, and is now supplying some items of this type, principally the pressure gassing equipment. This firm's experience has been principally in the packaging of pressurized food products, but has supplied some individual items of equipment for other types of aerosol filling lines.

Medicinal and pharmaceutical aerosols

By John J. Sciarra, Ph.D.
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An aerosol has been defined as a colloidal system consisting of very finely subdivided liquid or solid particles dispersed in and surrounded by a gas. The particles are generally between ten and fifty microns in size, sometimes even smaller. This definition proved satisfactory until about 1943 when the familiar "aerosol" insecticide was developed. This product depended upon the power of a liquefied gas (dichlorodifluoromethane) to produce a fine spray or mist of the insecticide. Therefore, through common usage the above definition has been modified to include all products which depend upon the power of a compressed or liquefied gas to expel the contents from the container irrespective of the size of the particles produced. In some instances, aerosols are referred to as "pressurized" products; especially those products dispensed as a solid stream, such as dental creams, vitamins, etc.

During the past several years thought was given to the utilization of the aerosol principle for the dispensing of medicinal and pharmaceutical aerosols. For the sake of clarity, in this discussion those products used for the administration of medicinals as

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fine solid particles or liquid mists via the respiratory system or nasal passages will be referred to as *medicinal aerosols*. These are generally intended for local action in the nasal areas, throat, and lungs, or for prompt systemic activity which is usually known as inhalation or aerosol therapy. *Pharmaceutical aerosols* may be defined as those products containing therapeutically active ingredients dissolved, suspended, or emulsified in a liquefied gas propellant or a mixture of solvent and liquefied gas or, as in the case of a compressed gas, in close contact with the propelling gas. Pharmaceutical aerosols are generally used externally as contrasted to medicinal aerosols which are used internally. However, there may be several products falling under both classifications.

Medicinals have been administered by inhalation or aerosol therapy for many years. The inhalation of smoke of asthma powders or the smoking of asthma cigarettes; burning of sulfur candles; and the breathing of salt air are just a few examples of aerosol or inhalation therapy. Since 1860 finely subdivided particles for inhalation therapy have been produced by atomization, vaporization, and nebulization, these all being mechanical means for the production of liquid mists.

Aerosol systems

The various types of available aerosol systems have been adequately covered in the literature. Those systems applicable to medicinal and pharmaceutical aerosols will be briefly reviewed.

Two Phase System—This system can be used for either medicinal or pharmaceutical aerosols. The active ingredients are dissolved in the propellant or a mixture of propellant and solvent. When the valve of this aerosol is depressed, active ingredients and propellant or propellant and solvent are released. The propellant and solvent will quickly vaporize leaving behind the finely subdivided active ingredients. While this system has been used to greatest advantage for both medicinal and pharmaceutical aerosols, several problems in formulation become immediately apparent. For the most part the therapeutically active ingredients are not directly soluble in the propellants and must be dissolved through use of a suitable cosolvent. The range of solvents available for this purpose is limited to those showing very little, if any, irritation and toxicity when used topically or by inhalation. Lack of adequate toxicity data on many solvents further complicates the formulation of such products. Ethyl alcohol, mineral oil, glycerin, propylene glycol, polyethylene and polypropylene glycol, acetone, and ethyl acetate (in certain restricted applications) are just a few of the solvents that can be used for this purpose. When one considers solvents for inhalation, the list is considerably narrowed.

Three Phase System—The therapeutically active ingredients are dissolved in an aqueous solvent and mixed with a relatively small quantity of liquefied gas propellant. The propellant (usually heavier than water) falls to the bottom of the container. When a hydrocarbon propellant is used (propane, butane, isobutane, etc.) the propellant, being lighter than water, rises to the top. By adjusting the length of the dip tube so that it does not dip into the propellant layer, when the valve is depressed only aqueous solution will be dispensed. By using a suitable actuator and baffle (similar to a nebulizer) the desired type sprays may be obtained. This system is currently under investigation for this application.

Suspension or Dispersion System—This system is used for those substances which are insoluble or can be rendered insoluble in the propellants. Through use of suitable dispersing agents the active ingredients are suspended in the propellant. In use, the propellant evaporates leaving behind the finely dispersed active ingredients. This system is finding application for medicinal and pharmaceutical aerosols and may well become the accepted method for the application of antibiotics and other medicinal agents.

Emulsified or Foam System—Through use of suitable emulsifying agents an aqueous solvent containing dissolved active ingredients can be emulsified with the liquefied gas propellants. While it is highly unlikely that this system will be used for medicinal aerosols, it is extremely useful for pharmaceutical aerosols. The product is dispensed as a foam which can then be applied to the affected area. In this manner the medicinal agent can be applied either to a limited area or to a larger area with very little, if any, irritation due to application of the medicinal. A recent advance in this area is the development of the "quick-breaking" foam. The product is dispensed as a foam but in contact with skin areas quickly collapses leaving behind the solution of medicinal agent. This makes possible the application of medicinals to wounded or abraded areas without the necessity of rubbing the medicinal to disperse it. This is highly desirable when applying medicinals to such areas.

Compressed Gas Systems—Nitrogen, carbon dioxide and nitrous oxide can be used as the propellant for medicinal aerosols, however the actuator and baffles must be such to produce a fine mist, similar to the three phase aerosol. The compressed gas acts as a piston to push the contents out the valve. Nitrogen is essentially insoluble in most substances while carbon dioxide and nitrous oxide show varying solubilities. Vitamins, dental creams, and some pharmaceutical ointments have been dispensed as a solid stream using nitrogen as the propellant. The preservative effect of nitrogen upon pharmaceuticals is well-known and has been established.

Nature of propellants

Most of the usual propellants can be used for medicinal and pharmaceutical aerosols. Propellant 11* generally shows high solubility characteristics but is the least stable. In the presence of moisture, hydrochloric acid is liberated which may cause a tingling sensation or cause irritation. It finds use in mixtures with Propellant 12 to lower the vapor pressure of Propellant 12. Greater stability to hydrolysis is shown by Propellant 114 making it useful in emulsion systems and other systems containing large quantities of moisture. Propellant 142b and Propellant 152a show promise for use in medicinal aerosols along

*For trademarks and chemical names of propellents, see the complete table elsewhere in this issue.

with Propellant 12. Propellant 152a is lighter than water and can be used in the three phase aerosol system. It generally is less prone to hydrolysis and causes less irritation than some of the other propellants. While it has a greater flammability it is less toxic. In medicinal aerosols, the quantity of propellant used is extremely small so that flammability ceases to be a problem.

Propellant C-318 shows promise of use in food aerosols as well as medicinal aerosols. Its toxicity is currently under investigation and has been found to be free of toxic manifestations to date.

The hydrocarbon propellants (butane, propane, and isobutane) are used in a similar manner to the fluorinated hydrocarbons. They are generally more flammable but less toxic, than the fluorinated hydrocarbons. Some may have a slight taste and odor which would have to be considered when used internally.

Medicinal aerosols and particle size

Particle size is an important consideration when medicinals are given by inhalation therapy. The site of deposition within the respiratory system, rate of dissolution, speed of absorption, and the efficacy of the preparation are controlled by the size of the particles. This subject has been covered in a report by Sciarra and Lynch. Several medicinal aerosols currently in use claim an average particle size of three microns in diameter with 75 percent of the particles less than five microns, 90 percent less than seven microns, and 99 percent less than ten microns. The intended use of the product will determine the desired particle size.

Since 1955 when "Medihaler" aerosol was introduced to the general public, many medicinals and pharmaceuticals were formulated into an aerosol type product. Table I lists some medicinal aerosol products while Figure 1 shows a display of medicinal and pharmaceutical aerosol products. The future may well see a tremendous growth of these aerosol products since they possess qualities not found in the usual type medicinal and pharmaceutical products.

Advantages of aerosol products

1—The product is more convenient to use, since the package is a compact unit and can be applied or administered easily and quickly.

2—Since the medication is sealed in a pressurized container, there is no danger of contamination of the product with foreign materials.

3—If the product is packaged under sterile conditions, then sterility is maintained throughout the life of the product.

4—The irritation produced by the mechanical application of a medicinal over an abraded area of the skin is reduced and sometimes eliminated by a "spray-on" aerosol.

5—In inhalation, the medicinal agent is absorbed directly into the blood stream through the alveolar membrane; therefore, there is less danger of decomposition of the medication, since it does not pass through the gastrointestinal tract.

6—Substances given by inhalation therapy are, in most cases, more effective than when administered by other routes, onset of activity approaches intravenous administration.

7—When drugs are administered in aerosol form, a high concentration of drug over a limited area can be obtained. This is especially useful in the treatment of pulmonary tuberculosis with streptomycin and other drugs.

8—Through the use of metered-dose valves, uniform dosage can be obtained.

9—Constant and a more uniform particle size is obtained compared to other methods of inhalation therapy.

10—Water-sensitive materials can be kept in an anhydrous state.

11—Pressurized powders can be conveniently used as insufflators for producing a finely divided cloud of powder.

Table I Medicinal Aerosol Products

Name of Product	Manufacturer	Ingredients	Use
Aero Meter	Rexall Drug Co.	Methapyrilene HCl Neomycin Sulfate Phenylephrine HCl Phenylpropanolamine	Nasal Decongestant
Medihaler-Iso	Riker Labs	Isoproterenol	Anti-asthmatic
Medihaler Ergotamine	Riker Labs	Ergotamine Tartrate	Migraine Headaches
Medihaler Epi	Riker Labs	Epinephrine Bitartrate	Anti-asthmatic
Medihaler Nitro	Riker Labs	Octyl Nitrite	Vasodilator
Medihaler Phen	Riker Labs	Phenylephrine HCl Hydrocortisone Neomycin Sulfate	Bronchodilator Nasal Decongestant
Nebu-Halant	Homemakers Products, Corp.	Contains no medication	Used as a nebulizer with separate medication
Mistometer	Winthrop Labs	Isoproterenol HCl 35% Ethanol	Anti-asthmatic

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Aerosol cleaning aids

By J. W. Baer
Industrial Consultant

Under this grouping we would like to discuss those pressure-packaged products which are generally purchased by the housewife for use in general cleaning chores. To gain the consumers' favor, these products must give an effect similar or better than those which are in common use in non-aerosol form. They must offer ease-of-use or some other feature such as safety to induce the user to pay the premium that the package generally carries over the established bottle or jar. To assure success for the household aerosol, a lot of thought and care must go into labeling design and label copy. Both the instructions and the cautions must be written in a clear, nontechnical manner. It is easy to frighten the potential purchaser if the product seems to be difficult to use. Many products have died on the shelves of the grocery or supermarket because the *Don'ts* were outclassing the *Do's* in the directions.

The chemical makeup of household aerosols often taxes the imagination and the patience of the formulator. It is next to impossible to take a product now successfully marketed in a conventional container and to convert it into an aerosol without a lot of reformulating. Often one has to develop completely new and different combinations to achieve the same end results. Let us look at a few specific groups of products presently on the market.

Window cleaners

Mixtures of water, alcohols, and wetting agents. While mild solutions of ammonia are successfully being marketed in non-pressurized form, the interaction of fluor-carbon propellents with alkali precludes their use in an aerosol can. Nonionic wetting agents such as the ethers seems to work best. Nitrogen pressurized window cleaners offer special low cost. This is an effective and easily made glass cleaner:

1.5	Silicone emulsion EF-4527
0.3	Triton W-30
3.0	Isopropanol 99%
0.3	Dowanol EE
0.05	Perfume
84.85	Deionized water
10.00	Propellent 12/114 60/40

Use an all nylon valve with stainless steel spring, epon coated cup, and mechanical break-up button. The can should be lacquer-lined, either 0.5 or 1.0 electrolytic tin plate.

In evaluating these sprays the writer found most corrosion to occur at points of stress created during crimping. By careful adjustment of the crimper's action this can be held to a minimum. Glycerin and glycols should be avoided since they often increase the corrosion problem. If the perfumery supplier is consulted, there should be no need to purchase a solubilized perfume, since the detergents in the formulation should take up the small amount of aromatic oils.

Dust mop sprays

Very useful products to reduce the spreading of dust during the daily wiping and to help maintain the satiny finish of furniture and wooden floors. It is an ideal example of how an aerosol product is a superior form of application. Various formulations are possible. The formulator should take care that the spray does not present a potential hazard by leaving the cloth or mop impregnated with chemicals prone to self-combustion. Hence mineral spirits, kerosene and naphthas should be avoided. Also a concentration of any latent solvents must be avoided to prevent injury to painted surfaces.

Formula I

20.0	Chlorothene
2.0	Mineral Oil White
0.5	DC–2000 Silicone Fluid 50cps
0.3	Perfume
4.7	Isopropanol 99%
62.5	Propellent 12/11 50/50

Use any suitable valve with a medium to fine spray delivery, such as 0.016-0.018 internal and 0.018-0.025 external orifices. The can we have selected is 0.5 tin lined.

Formula II

5 Emulsion of Silicones E-8
1 Solubilized Perfume
86 Deionized Water
8 Propellent 12/114 60/40

Use an all nylon valve, ss spring, epon coated cup, and a lacquer lined can. In formula II it is possible to use nitrogen as propellent by using a mechanical break-up button on the valve. While a metered valve might be handy, it is not necessary and would increase the cost of the final package.

Mothproofer & mothrepellent sprays

These products are very popular and are being marketed widely. They have to conform with all the provisions of the Federal Pesticide Act. Proven and approved formulation can best be obtained by consulting with the suppliers of the insecticide chemicals.

Among the preferred active chemicals are Strobane, Perthane, and Methoxychlor. The former two products are used in concentrates with deodorized kerosene, and from 4% to 5% actives by weight. Methoxychlor, which is preferred for carpet-beetles, is best used 1:4 with orthodichlorobenzene and paradichlorobenzene. The spray should be formulated for a wet, residual pattern, using 70% to 75% concentrate to 25%-30% Propellent 12, with a valve having a restrictive internal orifice with an external opening of 0.023-0.030.

The incorporation of cedar type perfumes gives the consumer an easily identifiable product, and offers a label advantage. The use of crude cedarwood oil gives lasting characteristics for cedarcloset sprays. In labeling one should call attention to thorough cleaning and brushing of all materials to be sprayed.

Garbage can sprays

Strobane, Dieldrin, and Chlorbisan are preferred active ingredients in this group of sprays. Since odors must be covered, good perfuming in addition to lingering killing power must be observed. Again, this product must conform to the regulations of the Federal Pesticide Act. Also the ultimate disposal of the garbage must be considered in the selection of the chemicals used to kill flies, maggots, and bacteria. In cities where the garbage is dumped or incinerated, no restrictions exist. If the product is to be distributed in rural areas where the waste foodstuffs are being used for hog feeding, Methoxychlor is preferred since it will not build-up in fatty tissue.

Formula I

1.5 Methoxychlor
.125 Chlorbisan
38.375 Petroleum distillates (Min. spirits)
0.375 Perfume
60.0 Propellent 12/11 50/50

Use a 0.5 tin plated can, and any medium fine spray valve.

Formula II

1.0 Strobane
0.05 Chlorbisan
73.55 Petroleum distillates
0.4 Perfume
25.0 Propellent 12 (100%)

Use a 0.5 tin plated can, and a medium fine spray valve, with a restricted internal orifice. As in any insecticide, the proper cautions must appear on the label regarding contact with eyes, skin, animals, foodstuffs, etc.

Fabric & rug shampoo

Detergent formulations have to be formulated with caution because of the great tendency to have detinning and corrosion occur in the can. Anyone who plans to market such a product must have undertaken extensive shelf-testing of his specific formula. Nonionic surfactants seem to work best.

We have tried various formulations with Sipon ES, and Nonic 300. A concentration of about 5%-8% of active with about 0.7% to 0.9% of lanolin esters gives a good foam which is easily worked into the material to be cleaned, and the excess is readily sponged off.

The consumer must be cautioned to test his material for color fastness; also in cleaning he should do several small consecutive areas rather than trying to do one large area at one time.

Spot remover

A product where many of the solvent type cleaning agents can be used without changing their formulation. Actually the propellents of the chlorinated fluorocarbon family are good cleaning agents themselves.

The important feature to observe in packaging these blends is that the valve parts, both the elastomers and the diptube must not be affected by the solvent. Neoprene gaskets and "rigid" polyethylene diptube are essential, and a nylon diptube is preferable.

Mineral Spirits (Stoddard Solvents) are not good ingredients for a spotting solution since they dry too slowly. The following is our preferred formulation. Good perfuming is essential to overcome the unpleasant odor of perchlorethylene.

25 Perchlorethylene
1 Perfume
74 Propellent 12/11 30/70

The use of a valve with a vaportrap to permit "upside-down" spraying is advisable.

Another approach to despotting is the use of a finely comminuted material as adsorbant. Such a powder, slurred in a solvent-propellent mixture is sprayed onto the soiled area, and after drying the powder is brushed out or vacuumed off.

10 Cab-O-Sil M-6
15 Perchlorethylene
0.5 Perfume
74.5 Propellent 12/11 50/50

Use a good powder valve such as the KN 38 with a 0.025 internal orifice. The usual cautions to the consumer as to testing for color fastness of dyes apply.

Aerosols in cosmetics and pharmacy, and compatibility test

By Dr. Otto Karl Jacobi
Kolmar Research Center

By aerosols we commonly understand a dispersion of minute solid or liquid particles in gas. In the first place air is considered a gas phase of the aerosols. Of course, there are also aerosols in other kinds of gas. Various types of aerosols very frequently occur in nature but also as a result of industrial or human activity in general. The size of particles of the dispersed solid and fluid portion of the aerosols respectively is decisive for their characterization. A most impressive comparison of the different types of aerosols was set up by Root (1). He classifies various well-known aerosols according to size of particles as follows:

Rain	100 to 1000 μ
Oil spray for oil burners	100 μ
Atmospheric nebula	2 to 15 μ
Tobacco smoke	0, 2 μ
Aerosol spray products	1 to 100 μ

This treatise will, however, be restricted to those aerosols which fall under the industrial definition of "a spray or foam product confined in itself in which the propellant force is represented by a liquefied gas or gas under pressure". Moreover, according to the definition of the subject only those products are to be dealt with which are produced and/or might be produced by the cosmetic or pharmaceutical industry or which are of interest to these industries.

For the formulation of aerosol products, four systems are available:

1. The three-phase-system: Here there exist beside each other the propellant, the concentrate of the product which does not intermix with the propellant, and the gas phase.
2. The two-phase-system: Here there exist beside each other the propellant in which the active substances of the product are dissolved and the gas phase.
3. The emulsion system: In this system the propellant is emulsified in an aqueous system.
4. The dispersion system: Here the active material is insoluble in the propellant mixture, but may be dispersed therein. This system is characterized by the powder spray aerosols.

The three-phase-system allows only very coarse sprays since the propellant actually serves as mechanical pressure potential only, so that merely the aqueous phase is dispersed.

The two-phase-system is the actual domain of the aerosols; however, it requires a solubility of the active ingredients in the fluid propellant. We know that in this system the fineness of the spray can be varied by selecting the propellants on the one hand and by admixing certain substances with the aerosol on the other hand. For instance, if the surface tension is reduced, the result will be finer sprays. Solvents with very low vapour pressure such as heavy mineral oils and silicon oils increase the average size of the aerosol particles, resulting in coarser sprays.

For the emulsion system Sanders (2) states that oil in water emulsions results in foaming aerosols while water in oil emulsions does not foam, producing much finer sprays.

According to Herzka (3) the use of a propellant entirely insoluble in the product is necessary for aerosol tooth pastes. The only fluid propellant which appears to meet with this requirement is octafluoro

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cyclo butane (Freon C 318). However, so far, at any rate in America, it is not admitted for use in edible products. The second best propellant for tooth paste aerosols, according to Herzka, is nitrogen.

The dispersion systems, or powder spray aerosols, create certain difficulties. The main problems are the development of suitable valves and the control of the size of particles.

In order to convey an idea of the development of aerosol products in the cosmetic and pharmaceutical industry, I am giving hereafter a survey of the American market published in July 1959.

In 1958, 178,841,000 aerosol packages were produced in the U.S.A., of which

51.8% were hair lacquers and liquid hair nets
25.3% were shaving creams
0.4% were sun tan products
7.6% were Eau de Colognes and perfumes
7.2% were tooth pastes
4.0% were pharmaceutical products
4.1% miscellaneous

For which kinds of products in the cosmetic and pharmaceutical industry will the aerosol package be suitable? The answer once was: Everything which can be sprayed is suitable for an aerosol package! But what is now actually produced as aerosols in the two industries of interest to us and/or considered possible as aerosol products?

Let us first view the cosmetic industry. The following preparations are already on the market as aerosol packages: room deodorants, air disinfectants, hair lacquers, liquid hairnets, aids for putting waves, shaving creams, anti-perspiration remedies, deodorants, perfumes, Eau de Colognes, toilet waters, hand lotions, sun tan preparations, shampoos, insect repellents, tooth pastes, skin lotions. More recently developed aerosols are: baby oil, baby talcum powder, products to prevent chafed skin, wound antiseptica, foam baths, nail varnish, mouth disinfectants, breath purifiers, body oil, talcum powder.

In the pharmaceutical industry the use of aerosols today is still relatively limited, but their range of applicabilities is very large, perhaps even larger than in the cosmetic industry.

What are the advantages offered by the aerosol package for pharmaceutical products, as compared to a regular package? The negative, often detrimental influence of light, humidity, and minerals on medicaments can easily be eliminated in aerosol packages. Very often the possibility of dosing is better. In preparations for external use an applicator such as a brush, absorbent cotton pad, finger etc. can be avoided, thus preventing loss and impurities of the medicament. Also the pains which are often caused when applying the medicament can in many cases be reduced, which is particularly obvious when treating burns. Moreover, it is possible to achieve a more even application on the wound. It is furthermore possible that medicaments applied in the form of aerosol can penetrate better. This, however, first needs to be proved.

E. Pomerantz (4) tested the durability of various medicaments in aerosols. He found ascorbic acid and thiamin to be more stable in a nitrogen aerosol than in a regular package. Contrary to expectations, however, vitamin A and B_{12} showed no increased stability in a nitrogen aerosol package. On the other hand, in foams and lotions aerosol boxes with halogenized hydrocarbons or nitrogen as propellant had no negative effect on the stability of vitamin A. The same applies for neomycin and hydrocortisone in an aerosol spray compound. Also the stability of a composition of neomycin hydrocortisone with polymycin in foams and lotions was influenced neither by halogenized propellants nor by nitrogen.

In this connection I may also mention an interesting and important diagnostic application of aerosols. It is a relatively simple and fast method of diagnosing cancer of lungs. The inhalation of minutely sprayed, warm propylene glycol particles causes the patient to eject sputum from spheres of the lungs which were previously unattainable. The microscopic investigation of the sputum reveals cancer cells if such are existent.

Today approximately 50 different pharmaceutical products are on the American market in the form of aerosol. Among those are: inhalation remedies for treating asthma, inhalation remedies for fighting colds, sprays for burns, local anaesthetics for skin and tooth treatments, antimycotics, sprays for treating skin irritations caused by a poisonous plant occurring in America (poison ivy), sticking-plaster sprays, etc.—Another group of products are sticking plaster removers, sprays for mouth and pharynx, nose sprays, antiseptics for external use, foot antiseptics, foot perspiration remedies, foot deodorants, drying, cooling, and astringent preparations for the treatment of the feet. Also a liquid multi-vitamin preparation for children was brought on the market in a nitrogen aerosol package. Another aerosol product is a dermatitis preparation.

On occasion of the Pharmaceutical Aerosol Symposium of August 20th, 1957 at the Philadelphia College of Pharmacy and Science in Philadelphia the following products were stated as having very good prospects in the aerosol domain: hormone, vitamin, antihistamine, as well as calamine preparations; zinc oxide, sulphur, resorcin, and tar preparations; remedies against itches, antipruritics, skin protection remedies, as well as cleansing, soothing, and smoothing lotions for the skin. Furthermore, the invisible aerosol compounds mixed up with dermatologic medicaments should be submitted to a test.

A domain which may offer a great future for pharmaceutical aerosols is the therapy of diseases of the ears, nose, and pharynx. Very little was done so far as regards the use of aerosols in the therapy of the ears and yet it should be possible to obtain a better penetration of the medicament in the middle ear region if it is applied in the form of an aerosol.

To a limited extent dentistry made use of the aerosols. Here they are employed as local anaesthetics, tranquillizers, astringents, and antibacteria remedies. There is great interest for aerosols as a prophylaxis in the cavity of the mouth.

The development of pharmaceutical aerosols for the possible use in treating rectal and vaginal states of diseases has found little attention so far.

Before dealing with the question of compatibility tests of aerosol preparations, we wish to make it

clear that we are not referring to the compatibility of the aerosols with the packing or of the propellant with the active substances, but to the compatibility of the aerosol products with the organ of application, i.e. the skin or the mucous membrane.

A finished product which is perfectly harmless in a regular box may lose this property in an aerosol container. This may be caused by the fact of the much finer dispersion and the creation of a many times enlarged surface as a result thereof. The finer dispersion causes a more intensive contact of the preparation with the organ. To a minor degree which, however, should not be overlooked, there are also toxic products playing a part, which may result from unforeseen reactions between the individual components of the product and the propellants.

When selecting the propellants, it is, of course, in the first place their non-toxicity which is decisive from the pharmaceutical and cosmetic point of view. The following products are known as being non-toxic: nitrogen, helium, argon, carbon dioxide. Of these, nitrogen probably will find a very wide range of application in pharmaceutical aerosols. The halogenized hydrocarbons which serve as propellant for the majority of the aerosols, may practically also be considered as non-toxic, as will be seen later.

In respect of the end products E. Pomerantz among others demands that toxicologic and skin irritation studies in any case be carried through before a new product gets on the market. Also Root (5) definitely points out the importance of testing cosmetic aerosol preparations in respect of toxicity and skin compatibility. With regard to aerosols, this point requires special attention inasmuch as aerosol dispersions produce vapor concentrations which do not occur when the same products are applied in conventional manner. In addition, the effect produced by inhalation should also be considered and tested most carefully. Besides the possible effects of incompatibility, there exists, however, another aspect which is again and again objected to by the consumers: The cooling effect on the skin when applying aerosols locally! How can this be avoided?

The quantity of the material sprayed onto the skin as well as the percentage and type of propellant exercize some influence on the degree of the decline of skin temperature. If the propellant evaporates partially in the air and not only on the skin, the decline of skin temperature is less. Consequently, Root (1) and Herzka (6) recommend for so-called warm sprays the use of propellants with a low boiling point. Contrary to this is the opinion of Mina (7), according to whom the effect of undercooling can be eliminated entirely by employing a high-boiling propellant such as dichlorotetrafluoroethane, but in smallest possible quantities.

How about the toxicity of the propellants themselves? According to Reed (8), the Freons—with which the German Frigens are identical, chemically as well as from the point of view of their nomenclature—as a group have a relatively low inhalation toxicity. This also emanates from the results of the investigations made by the Underwriters Laboratories published by Du Pont de Nemours & Co. Inc. For the purpose of testing the toxicity of various gases and vapors, guinea pigs for the duration of these investigations were exposed to breathing air containing different concentrations of those gases and vapors while their effect on the animals was studied.

Classified according to declining toxicity, the results of this range of investigations are as follows:

Sulphur dioxide	The effect of a concentration of ½ to 1% is lethal within 5 minutes or causes serious harm.
Ammonia and Methyl bromide	The effect of a concentration of ½ to 1% is lethal within ½ hour or causes serious harm.
Carbon Tetrachloride Chloroform and Methyle formate	The effect of a concentration of 2 to 2½% is lethal within 1 hour or causes serious harm.
Dichloro ethylene Methyl chloride and Ethyl bromide	The effect of a concentration of 2 to 2½% is lethal or causes serious harm within 2 hours.
"Freon 113" = $CCl_2F–CClF_2$	is less toxic than this last group.
"Freon 11" = CCl_3F "Freon 22" = $CHClF_2$	Are considerably less harmful than this last group. Their toxicity is equal to that of carbon dioxide.
"Freon 12" = CCl_2F_2 "Freon 114" = $CClF_2–CClF_2$	Concentrations of at least 20% do not seem to cause any harm within 2 hours.

Reed points out that this relatively low toxicity of the propellants may not simply be transferred to any finished aerosol product. Besides the effect of the individual components and their combinations, we may here possibly have to deal also with a special form of physiological activity caused by the extremely fine size of particles of the sprayed preparation. Horn (9), too, calls our attention to the risk involved in inhaling cosmetics in the form of aerosol sprays. He therefore recommends to carry through toxicological inhalation tests. The necessity of such an inhalation test may occasionally seem superfluous as a result of previous experiences with the same product or its components on the skin. Nevertheless, the same chemicals used in an aerosol formula may possibly cause serious injuries to the respiratory system. In this connection we must recall that the surface of the lungs is approx. 25 times as large as the surface of the skin.

Moreover, the lungs represent an extremely effective and fast means of absorption of chemicals. Furthermore, the deposit of fine particles in the terminal alveoli of the lungs may cause problems which are entirely different from those arising when applying the preparation on the skin. The surface of the lungs cannot be washed off like the skin. In other words, if the chemical gets into the lungs, it is deposited there and can no longer be removed. It is true that bronchia are equipped with a defense mechanism which, however, is no longer effective when the substances have reached the alveoli from where only absorption or enzymatic dissolution may accomplish a removal. If such a dissolution is not possible, the result may be a condition such as is known in silicosis of the lung.

In 1958 Bergmann and his collaborators (10) reported on 2 cases of lung affections which they traced back to particles of hairsprays deposited in the lung tissue. In experiments with animals which had received subcutaneous injections of hairspray, they found granules in certain tissues, which yielded a positive color reaction with per-iodic acid Schiff reagent. They assumed that these color-reactive deposits were macromolecular particles of hairspray. Calandra and Kay (11) tested the possibility of a lung affection by hairsprays on guinea pigs.

In inhalation tests they examined 2 branded products and 2 sprays produced by the authors themselves. Their tests furnished no proof of deposits of hairspray particles in the lungs. Granules susceptible to coloring with per-iodic acid Schiff reagent were also found in lung tissues of non-treated guinea pigs. However, what struck them during their experiments was the large percentage of animals which caught pneumonia. They presumed the cause of this to be the frequent temporary cooling off of the animals by their being exposed to aerosol. Comparative tests with animals which were repeatedly exposed to cold air and whose bodies as a result cooled off, showed the same frequency of pneumonia.

If hairsprays in fact should have a negative influence on the lungs, this would mean a public risk. Consequently Draize and his collaborators (12), officers of the U. S. Food and Drug Administration, submitted six different hairsprays to a most intensive inhalation test.

The authors examined sprays whose film-forming components belonged to the following chemical types: 1. Polyvinyl pyrrolidine, 2. a copolymer from polyvinyl pyrrolidine and polyvinyl acetate, 3. shellac, 4. dimethyl hydantoin formaldehyde resin, 5. lanolin, and 6. modified polyacrylic acid resin.

To begin with they ascertained with test persons the normal spray time which is 10 seconds, and the normal sojourn in an atmosphere saturated with hairspray, viz. 5 minutes. On an average, a test person exposed for 5 minutes to air saturated with aerosol inhaled 28 liters of air.

After these preliminary tests, they carried through their inhalation tests with rabbits. The authors preferred rabbits to guinea pigs because these animals are more resistant to cooling off which is unavoidable in aerosol experiments. They exposed the animals in a so-called acute inhalation test for 30 seconds at a time to an aerosol spray, which they repeated every half hour up to an overall number of 10 exposures. In a test lasting 90 days the animals were exposed to an aerosol spray twice daily for 30 seconds each and remained 15 minutes in the aerosol atmosphere. The lungs of the animals submitted to the acute test as well as those of the 90 day-test were examined rontgenologically, blood tests were analysed and autopsies as well as histopathological examinations were carried through. The authors could not ascertain any alterations of the lung tissue. They therefore came to the conclusion that aerosol hairsprays in those compositions and physical characteristics as they were tested do not hold any danger for the consumer in normal use.

The general methodical directives published by the Food and Drug Administration for the examination of the security of chemicals employed in foodstuffs, pharmaceutics and cosmetics urgently recommend to test aerosols, and this not only by submitting them to the inhalation test. The publication states that in addition to the possible injuries of the skin and mucous membrane, aerosol preparations hold toxicological problems of inhalation. Particles of 3μ and less than that easily reach the alveoli of the lungs and while particles of 3 to 10μ reach the parenchyma of the lungs with considerable difficulty only, those of over 10μ will hardly penetrate into the tissue of the lungs. However, as is well-known, the size of particles depends on the aerosol system, the mixture of the propellant, as well as on the valve. Therefore, new aerosol preparations should not only be submitted to inhalation tests, but to toxicological examinations in general, same as all the new preparations to be brought on the market.

We will now show a few test possibilities for products to be applied externally, which are of prime interest to us. Quevauviller and collaborators (13) recently proved on rats the good skin compatibility of various chlorofluoro methanes. They examined CCl_3F, CCl_2F_2 and $CClF_3$, each by itself, in a mixture, and in oily solution. Applying it 3 times daily for 2 months, they did not notice any alterations of the skin.

Genuine Irritation of the Skin: A substance causing a reaction at the first contact with the skin may be defined as a genuine irritant of the skin.—If the skin is able to cope with the irritant, the reaction is physiological. However, if the nature of the attack exceeds the normal powers of defense of the skin, the reaction is pathological and the result is an irritation.—A genuine effect of skin irritation will be tested by means of the technique of applying small rags on the depilated, intact, and mechanically injured skin of the rabbit.

The Sensibilisation Test: Skin sensibilisation is a phenomenon revealing itself by local effects on the skin or the mucous membranes. It differs from the genuine irritation of the skin in that it does not occur at the first contact. To that effect a repeated contact will be necessary. It may also affect tissue far remote from the place of application. The number of contacts with a substance, which are necessary in order to release the reaction, may vary considerably. It depends on the sensibilisation potential of the substance as well as on the individual.

In order to recognize strong sensibilizers, the intracutaneous technique proved best on guinea pigs. To that end an intracutaneous injection with a solution of the substance to be tested is given every other day over a certain period of time, up to a total of 10 injections; 2 weeks later the release injection is made. The skin reaction visible 24 hours later, in relation to the reactions of the first injections, serves as criterion for the sensibilisation.

This test as well as all the other animal tests, of course, have to be modified if necessary in accordance with the product to be tested. Skin irritation tests and sensibilisation tests are also carried through on human beings. However, the rag test on rabbits and the intracutaneous test on guinea pigs should always be carried through before any test is made

with man in order to eliminate from the beginning heavily irritating substances and to thus avoid exposing human beings to serious irritations of the skin and sensibilisations. When developing new products, which is always done in large series, the animal test is the means for ascertaining detrimental individual components and combinations.

In order to investigate on the genuine irritation of the mucous membranes, we use in animals the vagina (preferably of dogs), the penis, and the conjunctiva sac of the eye. For the eye test mostly rabbits will be employed.

In order to ascertain injuries of inner organs as a result of penetration through the skin, the preparations will be applied to major regions of the skin of the test animals, one or several times, whereby applications will be made daily up to 90 days. At the same time these tests give an opportunity of studying the compatibility of the skin over prolonged periods of time. During the entire period of the tests the general behavior of the animals, their weight, and the quantity of food consumed daily are regularly supervised. Also urine and blood are currently examined. Finally those animals which succumb during the tests or suffer from serious effects of poisoning are opened and the organs are examined with respect to pathological and histopathological alterations.—Such experiments yield most valuable data as regards the reaction of a product in permanent use, especially since the duration of the tests may be prolonged at will.

Summary

After a brief characterization of aerosols and aerosol systems, we have described cosmetic and pharmaceutical products in aerosol containers, which are already on the market or which may still appear. Furthermore, we have shown the toxic effects which possibly may be caused by aerosol products. Although propellants are generally regarded as harmless, in the case of aerosol preparations the fine dispersion and the consequent large surface with which the materials contact the organs create unusual conditions. Increased activity and also an increased potential of irritation may result. Additional irritants may develop by collateral reactions of an unknown nature between the propellants and the chemicals employed in the formulas. The compatibility test of finished aerosol products has been described by means of several examples.

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Vapolytic finish is a new development by Crown Cork and Seal Company. This is a brilliant metallic finish put onto Crown's six ounce Spra-tainers. It is a vacuum metallizing process, and provides a wide variety of metallic colors with the luster formerly only available through the use of foil labels.

Room deodorants and sanitizers represent a field that depended upon the aerosol package development for important growth. The previous type of room deodorants, in wick type packages, were very slow sellers. With the development of the aerosol package for these products, sales have spiraled upward, and as yet show no sign of leveling off.

Extruded, seamless aluminum aerosols have been developed by Impact Container Corporation in sizes from three ounces to thirty seven ounces. The thirty seven ounce container weighs only four ounces. 300 psi is the standard burst strength, and 600 is also available. Epoxy interior coatings are also available.

A summary of a talk presented at the Seminar on Aerosol Technology, St. John's University. The paper was published in **Aerosol Age**, June and July, 1960.

New drug applications for aerosols

By Julius Hauser

Food and Drug Administration

An expert on aerosol drugs should be familiar with the preparation of new drug applications.

The manufacturer of a drug is responsible for its compliance with the Federal Food, Drug, and Cosmetic Act and should determine that his product is not adulterated or misbranded and is not in violation of the new drug requirements. Comment on the legality of a proposed product may be obtained by submitting its complete composition and proposed labeling to FDA. Time and expense may be saved by submitting an inquiry rather than a new drug application on an aerosol that may not be a new drug.

The Act prohibits interstate shipment of a new drug until an application is effective for it. The application must satisfy the Food and Drug Administration that the new drug is safe.

Labeling as well as composition determines whether an article is a new drug. An aerosol may be a new drug if it contains a novel active ingredient, solvent, or propellent; is a new mixture of old drugs; is intended for a novel use or method or duration of administration or application; or because of an inhalation hazard even though it is not offered for inhalation use.

A written opinion from FDA that an article is not a new drug insures against litigation on a new drug charge. A correspondent seeking such an opinion should show that the drug is one generally recognized as safe by experts and has been used to a material extent other than in investigations of its safety.

A new drug application should contain (1) full information on safety studies; (2) components of the drug; (3) quantitative composition; (4) manufacturing methods, facilities, and controls; (5) required samples and; (6) proposed labeling.

It is the policy of FDA in processing applications to employ the concept of safety as broadly as possible to insure the efficacy of new drugs. It may be futile to submit a new drug application for an article that is worthless or for which there is no evidence of usefulness.

A new drug application form available from FDA furnishes a detailed outline. If able scientists have satisfied themselves of the safety, usefulness, integrity, and stability of the new drug before assembling the application, there will probably be little difference of opinion before favorable action. An application that does not satisfy the applicant's own scientific staff is likely to encounter considerable difficulty and delay.

It is not possible to design a single protocol of the studies that are necessary and sufficient to establish the safety of all new drugs. Such investigations are related to the nature of the article and its intended conditions of use. FDA scientists cannot make advance commitments but will furnish comment on proposed plans of study. A booklet, "Appraisal of the Safety of Chemicals in Foods, Drugs, and Cosmetics," published in 1959 by the Association of Food and Drug Officials of the United States, contains FDA views on safety studies. Of particular interest to aerosol manufacturers is the chapter on "Dermal Toxicity." Tests frequently applicable to aerosols include inhalation toxicity, primary irritation, and sensitization on skin and mucous membranes of animals and man, and bacteriological characteristics.

The data in a new drug application may be submitted directly by the applicant or incorporated by an authorized reference to pertinent information submitted by the applicant's supplier of raw materials, containers, valves, or manufacturing or control services.

Among the controls frequently required for aerosols are specifications and batch tests with respect to pressure, leakage, bursting hazard, valve function, spray characteristics, particle size, bacteriological quality, and the quantity of product delivered by metered valves. Control should also determine the actual quantity delivered by a pressurized container and the label statement of quantity should not exceed the minimum delivered. Stability studies of pressurized aerosols should show not only that the drug is stable but that the container and valve remain sound and in good working order.

The labeling of an over-the-counter aerosol should include any applicable warning statement in the list published in the *Federal Reigster* of March 25, 1960. The label of a pressurized container should include the warning statements required by the Interstate Commerce Commission regulations and may also require a warning with respect to the distance from which it should be applied to the body to avoid freezing.

Full information for professional use should be included on or within the package of an aerosol prescription drug. This may require a quantitative statement of both composition in the container and composition as it reaches the body. It is not required and may be unnecessarily costly to prepare final printed labeling for initial submission with a new drug application.

The "New Drug Regulations" available from FDA answer other questions on applications.

Setting up an aerosol laboratory

By William Scheck

Builders Sheet Metal Works, Inc.

Have you a product that you would like to put into an aerosol package? If so, your firm is just one of several thousands that have done so, or are seriously thinking about it. The variety of items that might be adaptable are almost limitless, and this unique type of package might be just the thing to rocket your sales.

There are several good reasons why you might want to start your own aerosol laboratory for development work, rather than depending on any one of the many good independent contract loaders. These reasons are:

1. The time saved traveling back and forth with samples from your contract filler will enable you to put the product on the market sooner.
2. The contract filler may be too busy to give you the benefit of his laboratory and experience.
3. You may want to expand your own knowledge of this method of packaging.
4. A product is to be developed which requires close supervision or is of a secret nature.

Sources of information

To learn as much as possible about a laboratory set-up write to the suppliers of propellents for their literature. The four producers of the fluorinated hydrocarbons, the primary propellents, are duPont, General Chemical, Pennsalt and Union Carbide. Their addresses are given at the end of this article. There are other firms who publish literature on this subject, and they are also listed in the literature references. There are also a number of trade magazines that give coverage to new developments in the field of aerosol packaging.

Visit aerosol laboratories

One of the finest ways of determining the type of set-up and equipment that you need is by visiting existing laboratories. This can be especially useful when visiting suppliers with this type of set-up, as they can, and usually will, be most helpful in answering questions and suggesting the best way for you to proceed.

Equipment required

After visiting laboratories and studying the literature, the following pieces of equipment are probably what you will find are necessary for a complete laboratory set-up.

A pressure filler for forcing the propellents into your containers at room temperature to provide the propelling force.

A "cold" filler which cools the propellents below their boiling point and enables you to pour the cooled liquids into the container which also must be pre-cooled. When the sealed container reaches room temperature the propellant builds up enough pressure for dispensing of the product. Some delicate odors and flavors are affected by low temperatures so these items must be pressure filled.

A valve crimping device preferably for both cans and bottles.

Pressure testing devices to determine pressure of containers.

A *nitrogen pressurizing unit* for use with food and viscous products.

Compatability bottles which enable you to examine the products for precipitation and discolorization.

A *water bath* held at approximately 130°F. to test the containers for leaks by watching for bubbles. If the container bursts there will be little damage as the water acts as a shield.

A corrosion test oven which is held at approximately 130°F. The primary purpose of this cabinet is to determine the shelf life of a pressure propelled product before it is placed on the general market. When the test has been completed, the container is cut apart and examined for evidence of corrosion, changes in the product etc. Approximately one month in the cabinet will equal 8 or 9 months on the dealer's shelf.

All this equipment should be in an area a minimum of 6 ft. x 12 ft. and provisions should be made for work table space, tank racks for the inverted propellent tanks and a sink for washing and disposing of unwanted items.

Safety rules

The following general safety rules should be carefully and completely followed at all times:

Do not heat aerosol containers except under carefully regulated conditions which minimize the danger of bursting.

Do not close any 2 valves in a full liquid line as the possible expansion of the liquid due to a temperature change can rupture the line and lose the propellent.

Do not overfill containers as the same thing can happen. With little or no head space the liquid expands and exerts terrific hydraulic pressure and breaks the container.

Formulas should be carefully considered so that no items when mixed will create a pressure in excess of the container capacity. Toxicity and flammability must also be considered.

Adequate ventilation should be provided as propellents can blanket out the oxygen if released in large quantities in a small area. Ventilation is especially important in propane and butane experiments as pockets of these gases can form where least expected if the area is not properly ventilated.

Eye shields are used by a good number of lab technicians when the operation has a special hazard.

Now that you have started, you become a part of one of the fastest growing industries in this country. If all goes well with your products, profits should be higher and your knowledge increased, both of which should give you a smile of pleasure.

Publications of interest to aerosol laboratories

"Package for Profits", DuPont Co., Wilmington 98, Delaware.

"Kinetic" technical bulletins, DuPont Co., Wilmington 98, Delaware.

"How to Push Up Profits with Aerosols", General Chemical Co. Div. of Allied Chemical, 40 Rector St., New York City.

"Basic Aerosol Formulations", Pennsalt Co., 3 Penn Center, Philadelphia 2, Pennsylvania.

Bulletins in relation to aerosols, Ucon Div. of Union Carbide Co., 30 East 42nd Street, New York 17, N. Y.

"Get Bigger Profits from Pressure Packaging", Crown Cork & Seal Co., Philadelphia 36, Pennsylvania.

"Facts on Gases Used for Food Products", Air Reduction Co., 150 East 42nd St., New York 17, N. Y.

"The Aerosol Story", Dodge & Olcott Co., 180 Varick St., New York City.

"Aerosols and Pressurized Products", McLaughlin Gormley King Co., 1715 SE 5th St., Minneapolis, Minn.

"Aerosol Laboratory and Production Equipment", Builders Products, 108 Wooster St., New York 12, New York; Colton Alpha Co., 3698 E. Lafayette, Detroit 7, Mich.; Kartridge Pak Co., 9151 Fullerton Ave., Franklin Park, Ill.

"CSMA Aerosol Guide", Chemical Specialties Manufacturers Association, 50 East 41st St., New York City.

Aerosol publications, Givaudan-Delawanna Co., 321 West 44th St., New York 36, N. Y.

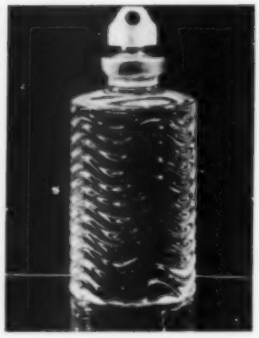
"October Buyers' Guide", Aerosol Age, Box 31, Caldwell, New Jersey.

"Blue Book of Soap and Chemical Specialties", McNair Dorland Co., 254 West 31st St., New York 1, N. Y.

"Pressurized Packaging (Aerosols)" by Herzka and Pickthall.

"Formulations of Cosmetics and Cosmetic Specialties" by W. R. Keitler.

Valve and container information and samples from selected manufacturers.



Uncoated glass aerosols have been quite rare in this country, though quite common in Europe. This container, by Par Industries, Inc., with intricate convolutes has just been introduced. Jointly developed by Par Industries, and Old Empire, this smart cosmetic bottle holds 1½ ounces, and is available with several types of overcaps that cover the valve and fit flush with the top of the bottle.

Perfumed cream soap is being marketed by Angelique in their four Satin fragrances, in aerosols. The main selling feature of this form of soap is that it can be formulated with much higher percentages of lanolin than bar soap. It is claimed that this soap has much of the effect of both a soap and a lotion.

Plastic coated glass bottles, by Wheaton Plasticote Corporation. The range and style of these bottles indicates that the variety of shape, size and type of color and finish is almost unlimited.

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SPEAKING FROM THE U.S.A.

ROBERT P. T. YOUNG, B. CH. E., M.S. IN ENG., *was formerly Section Head in charge of Dentifrice Process Development, Basic Development, and Perfumes and Flavors for the Toilet Goods Division of The Procter and Gamble Company. He was with Procter and Gamble since 1941 with the exception of the six years he spent in the Navy—During World War II he was concerned with the design, manufacture, and inspection of 40 mm. ammunition; during the Korean war—propellants for rockets and missiles. A lieutenant commander, he was Head of the Test and Quality Control Division, Naval Powder Factory, Indian Head, Maryland. He received a Letter of Commendation for his contribution to the design and development of the Mark 43 solid propellant rocket grain.*

Glycerine IN DENTIFRICES

Benjamin Franklin once wrote, "Many complain of their memory, but few of their judgment." Since we can assume straight-off that readers of this article are people of good judgment, it seems quite proper to offer a memory-aid, or key-word, to use in considering the role that glycerine plays in modern dentifrice formulation. The key-word for a dentifrice formula is *BASHFUL* and all dentifrice ingredients can be classified into one of the following groups (remember the key-word):

B is for binders

A is for abrasives

S is for sudsers

H is for humectants

F is for flavors

U is for unique additives

L is for liquids

Glycerine is a humectant, and as you will see it has a rather central position in a good dentifrice formula. A humectant can affect every other ingredient, and its presence in the formula is, I believe, the principal reason the American people overwhelmingly prefer toothpaste to tooth-powder. It may be of some interest in passing to note that the largest selling dentifrice in America (1959 figures) has the highest glycerine content.

Binders

B is for binders and binders stabilize the paste. All binders provide highly viscous aqueous systems, and the best are quite thixotropic or gelatinous in nature. The purpose of these stabilizers is to prevent the separation or stratification of the various ingredients used, and when one considers the diversity of these ingredients—from heavy, solid, inorganic abrasives to light liquid, organic essential oils—one realizes the magnitude of the binder's task.

One of the earliest binders used was glycerite of starch. This was followed by various natural gums: karaya, gum arabic, or gum tragacanth. Today, the principal attention by dentifrice formulators is directed toward seaweed derivatives (Irish Moss) and toward the synthetic celluloses. (1) In addition to these organic binders, bentonite, a native, colloidal, hydrated aluminum silicate, and Veegum, a complex colloidal magnesium aluminum silicate, have been recommended for use. (2)

Almost every edible thickening agent can be considered to be a potential dentifrice binder. A simple screening test to determine the potential usefulness of a material as a dentifrice binder is to make a 1% solution of it in a 50:50 glycerine-water solution. If the binder is incompatible in this system (and many natural materials such as locust bean, guar gum, etc. are), it is not likely to be a very satisfactory dentifrice ingredient.

Gershon et al. (2) mention that one of the best ways to make a toothpaste is to disperse the binder first in the humectant and then add this slurry to the aqueous phase. In this type of processing an essentially anhydrous system is needed for best dispersion, and glycerine is ideal.

In choosing a binder, particular attention must be paid to its interaction with all other ingredients. Since most abrasives contain trace quantities of cations (calcium or magnesium ions), these can interact at available sites on the binder molecule and cause firming or lumping on long-term storage. Some binders, particularly Irish Moss extractives, act synergistically with the sudsers to produce a more stable foam on brushing. It is necessary to balance the binder with the humectant to arrive at the ideal viscosity, and also to produce a glossy, short-textured product. Finally, the binder must be completely bland in flavor and odor, and it must be compatible with the unique additives used in the formula.

Abrasives

The purpose of dentifrice abrasives is to remove the stains which form on teeth. Ideally, these materials should accomplish this without damaging the soft part of the tooth structure (dentin) which is often exposed near the gum line.

The literature covering dentifrice abrasives is most fascinating. Historically, such exotic materials as pulverized eggshells, rouge, charcoal, wood sawdust, silicon carbide, and powdered cuttlebone have been mentioned. (3) The materials recommended for use today are much more commonplace consisting of :

Dicalcium phosphate
Tricalcium phosphate
Calcium carbonate
Magnesium hydroxide
Diammonium phosphate
and similar materials

Kuever (4) patented the use of insoluble sodium metaphosphate. Phillips and Van Huysen (5) reported that when this material was mixed with calcium phosphate some polishing action occurred. Special abrasives have also been designed for special purposes.

The interaction of humectants with abrasives is quite important to the "mouth-feel" of the dentifrice. Great care must be exercised to secure the proper balance between "chalkiness" on one end of the scale and "sliminess" on the other. Interestingly enough, there is a profound difference between humectants in this respect and glycerine seems to offer more flexibility in formulation than the others.

It is known that glycerine reduces the abrasiveness of the abrasives used without substantially affecting their cleaning power.

Sudsers

Longfellow (7) reports that soap has been replaced by synthetic anionic surface active agents in all the major brands of dentifrice. The principal sudsers in use today are:

Sodium lauryl sulfate
Sodium N-lauroyl sarcosinate
Sodium lauryl sulfoacetate
Dioctyl sodium sulfosuccinate
Sodium salt of sulfonated coconut monoglyceride

All of these materials must be highly purified to prevent imparting undesirable (mostly bitter) flavor notes to the dentifrice.

Citrus fruit and other acid foods tend to have a bitter taste if eaten soon after the teeth are brushed with a dentifrice containing a synthetic surfactant. (8) Sanders (9) has obtained a patent on blends of sodium lauryl sulfate (1 part) with monoglyceride sulfonate (2 to 5 parts) which are quick sudsing and have no more effect on the taste of orange juice than the pure monoglyceride sulfonate.

One of the differences between soap and synthetic surfactant is the feel or "slip" of the foam produced. Over the years people have become accustomed to the soft, slippery feel of soap, and the synthetics seem harsh or "squeaky-clean." Glycerine does much to overcome this objection—foams of systems containing synthetic surfactants are markedly smoother and more stable if glycerine is added.

Humectants

The principal purpose of using humectants in a dentifrice is to prevent the paste from drying out. The most serious type of dry-out occurs at the tube nozzle particularly when the cap has been removed and lost. Most pressure-packed formulae are particularly high in humectant level since normally no cap is used and because the polyethylene nozzle is rather readily permeated by air and water vapor.

When considering humectants for dentifrice only three materials are usually mentioned:

Glycerine **Propylene glycol**
Sorbitol

Propylene glycol is reported to have a slightly acrid taste. (10) It has not been widely used.

In considering the relative merits of glycerine or sorbitol as humectants for toothpaste, it is fair to state that glycerine offers all the advantages of a near anhydrous liquid. Sorbitol, which is a solid when anhydrous, tends to produce firmer pastes. Glycerine also has the tremendous advantage of being the traditional humectant used, and its interaction with all of the other ingredients in a dentifrice is established and well-known.

Flavor

One of the most important consumer properties of a dentifrice is its flavor. The flavors of the leading dentifrices in the American market are fundamentally mint, wintergreen, or root beer in type. A dentifrice flavor consists of volatile essential oils (peppermint oil, spearmint oil, methyl salicylate, menthol, anethole, etc.) and crystalline components—saccharin, cyclamate, salt, etc. The base stock also contributes flavor, and the most advantageous of the ingredients in the base stock, from a flavor point of view, is the glycerine. Carr, Beck, and Krantz (11) have furnished the following table:

Relative Sweetness of the Sugar Alcohols
(Sucrose Taken as 100)

Glycerol	108
i-Erythritol	238
Pentaerythritol	110
d-Mannitol	57
d,-l-Sorbitol	54
i-Dulcitol	74
Inositol	50

From the above, it appears that glycerine is twice as sweet as sorbitol. Mannheim (12) reports that a very popular dentifrice used in Europe during the '30s contained 38 per cent glycerine and required no, or very little, saccharin for sweetness.

Unique Additives

Therapeutic dentifrices have received increasing attention during the past ten years. They owe their performance to ingredients we have termed "unique additives."

The list of unique additives is a long one. Those intended to reduce caries have included:

Ammonia salts and urea compounds
to neutralize mouth acids.

Sodium N-lauryl sarcosinate
to act as an anti-enzyme.

Sodium oxalate
to build an insoluble film on teeth.

Hexachlorophene
to destroy mouth bacteria.

Stannous fluoride
to reduce the solubility of tooth enamel in acid. (13)

Tyrothricin
to act as a general antibiotic.

The search for unique additives has even gone as far as Ora S. Flower's (14) idea of adding living organisms, *Bacillus alcaligenes*, to the paste and thus inoculate the mouth with alkaline-producing bacteria.

New Zealand has the highest incidence of dental caries in the world and Drs. Irwin and Leaver (15) have clinically tested tetradecylamine acetate there.

Unique additives have been recommended for purposes other than reducing tooth decay. Mouth odor is a particularly important problem, and toothpastes green with chlorophyll were once in vogue. It is sometimes necessary to add a preservative to protect the paste itself from microbial attack and ethyl p-hydrozybenzoate at 0.1 per cent of formula has been recommended. (16) The removing of stain from the teeth by the addition of dextran to the formula was recommended by Russell and Nelson. (17)

Glycerine is a particularly valuable ingredient in a dentifrice when its interplay with the unique additives is considered. Many of these materials are reactive chemicals and more or less soluble in water. Instability of these actives through chemical reactivity of one type or another becomes a serious problem as the pastes age. One of the best ways to reduce this reactivity is to replace as much of the water as possible with glycerine.

Liquids

The common liquid in all dentifrices is water. Deionized or distilled water is recommended to minimize the possibility of color or flavor changes.

Small amounts of mineral oil have sometimes been used, principally to make it easy to extrude the paste from the tube. It is quite obvious that mineral oil puts a tremendous load on the sudsers.

Humectants and water make up the large bulk of the liquid phase of the dentifrice. The relative amounts used have a marked effect on the physical as well as the brushing properties of the paste. A good place to start in designing a new formula is 50:50 glycerine-water.

SUMMARY Using the key-word, BASHFUL, the formulation of a dentifrice has been discussed. It has been shown that glycerine interacts with each of the other ingredients, and that it is indeed an important and beneficial component of a fine dentifrice.

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NOTE: Inclusion of formulations in this series of articles does not constitute a recommendation by G.P.A.—nor warrant that they are free from patent or other legal restrictions.

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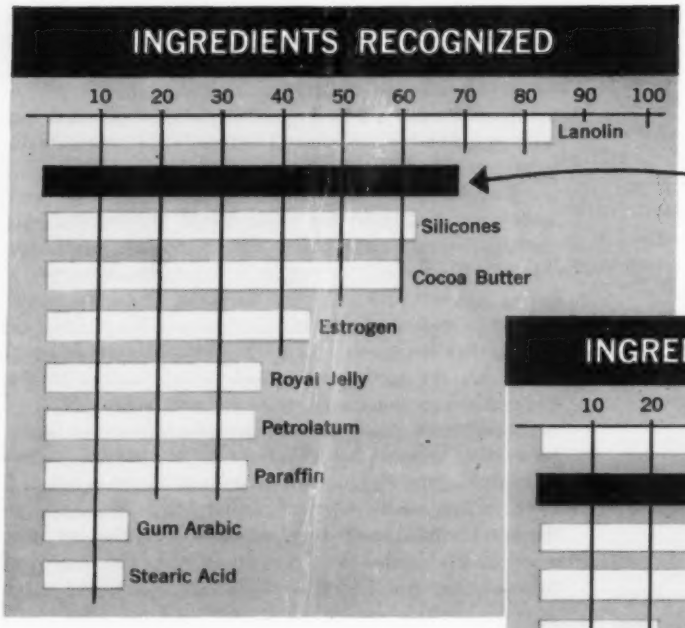
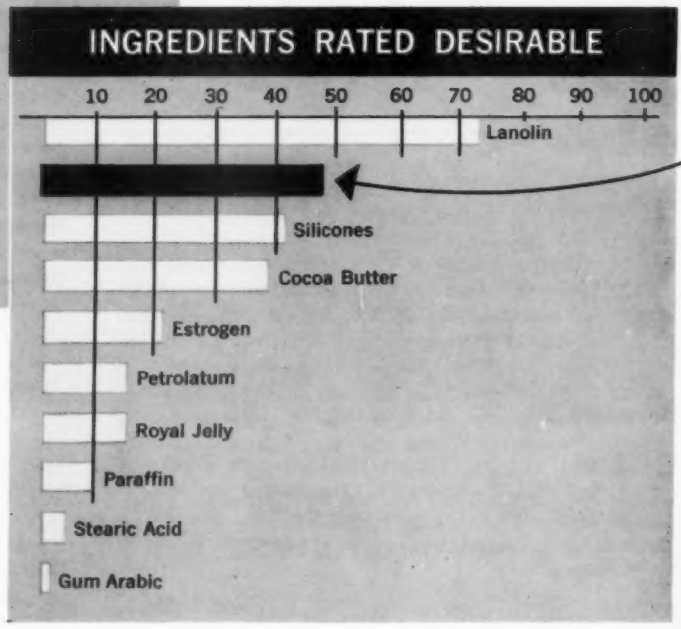


GLYCERINE



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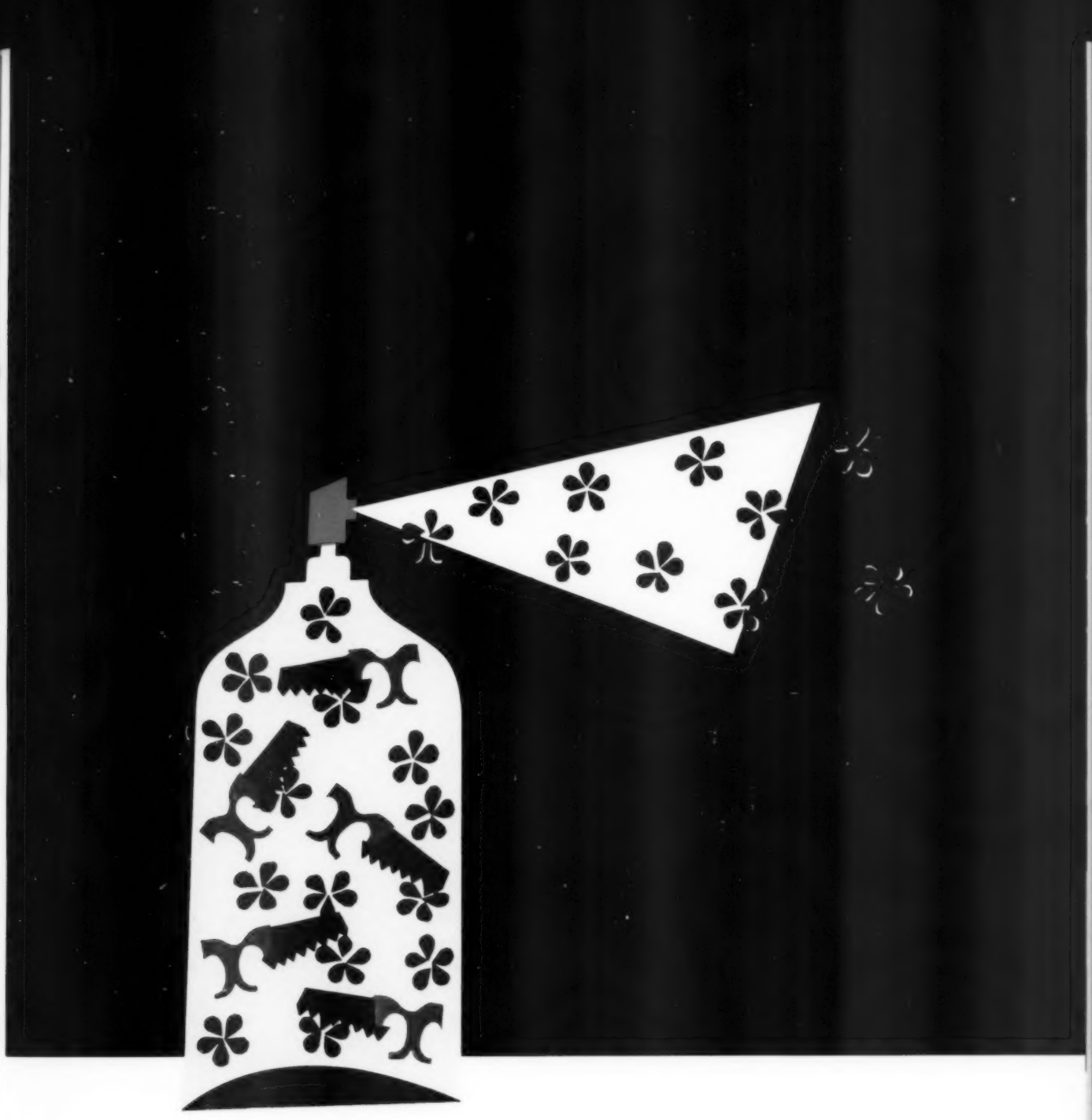

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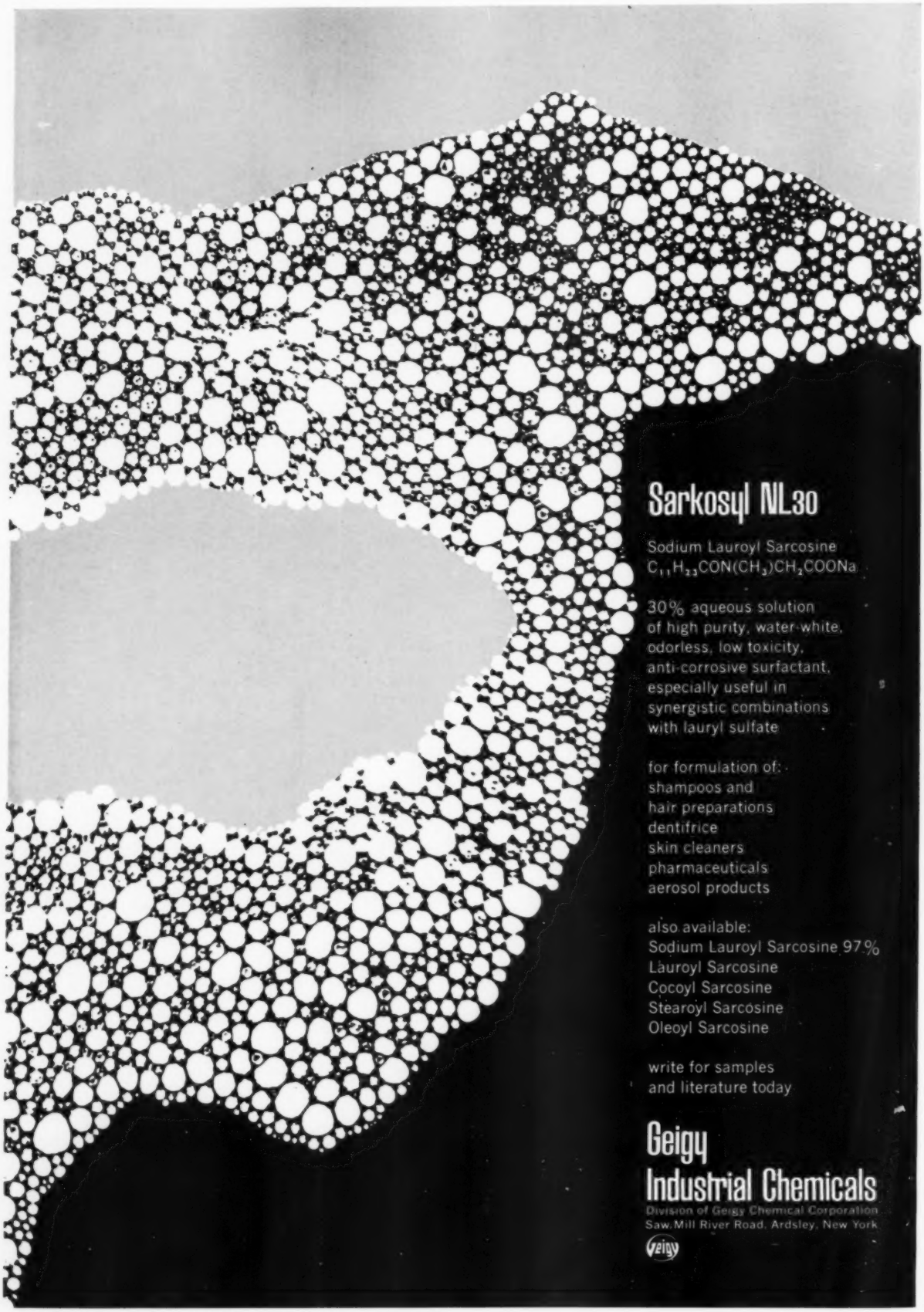
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odorless, low toxicity,
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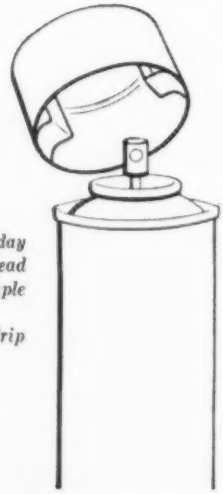

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Cold versus hot fill process

By Robert A. Foresman, Jr.
Aerosol Consultant

For those not thoroughly familiar with the two processes at issue in this article, it may not be amiss to describe them briefly. The terms cold fill and hot fill refer to the two methods in use for adding liquefied propellents to aerosols, or more properly, pressure packages.

Liquefiable propellents can exist in the liquid state under two conditions—either at pressures above their vapor pressure or at temperatures below their boiling point.

Cold fill refers to the process whereby the propellent (as well as the concentrate) is chilled by refrigeration to below its boiling point and handled and filled as a chilled liquid at atmospheric pressure.

Hot fill, or more accurately pressure fill, refers to the process of maintaining the propellent under pressure at ambient temperatures. The propellent is handled in a closed system as a liquid under pressure throughout the entire process, including that of filling it into the container.

In passing, we might mention another type of pressurized package which really lies outside both of the processes we are concerned with here. These are the products gassed with compressed or soluble gases. Here the gases are introduced into the container as gases in the vapor state and at room temperature. The quantity of gas introduced is simply a function of temperature and equilibrium pressure between the source and the inside of the container. Since agitation coincident with gassing is usually desirable in order to achieve equilibrium in minimum time, specialized equipment is used which is neither cold nor hot fill as those terms are used here.

Although nominally this is a discussion of cold fill versus pressure fill of aerosols, there is currently very little justification for considering cold fill for new installations.

The cold fill process is still a factor in the aerosol industry for only one reason—the existence of large investment equipment that will still perform satisfactorily and has not been paid off to the point where its owners feel it can be jettisoned economically.

Historically, the reason for the early popularity of cold filling was simply that equipment for the handling of liquid at atmospheric pressure and low temperature was easiest to develop and more readily made available to the industry than the more mechanically involved but more efficient pressure filling equipment.

One reason that cold fill equipment held sway for so long was the greater speed of which it was capable. Large volume fills of chilled propellent could be gushed through the uncapped mouths of aerosol containers in much less time than the same amount of propellent could be pushed backward through the small opening of the container valves after the caps were in place. This comparative condition continued to exist until engineering advances by the equipment and valve manufacturers made high speed pressure filling possible.

The first approach to increasing the productive capacity of the pressure fill process was to develop valves especially for such use. Conceptually, of course, this required a valve incorporating all the minuscule dispensing orifices present in the conventional valves for flow from inside the can to the outside, combined with large unobstructed passages for free flow in the opposite direction. As these valves became available and production at a practical level became possible, pressure filling began to develop popularity.

Equipment manufacturers turned to the design of pressure filling equipment as the ultimate answer to making aerosol filling a practical matter for the packaging industry at large. In the course of time high speed multiple head units were made available. These consisted of both straight line and rotary type machines which made speeds in the range of one hundred units per minute (of large volume fills) possible.

Finally, and very recently, the under-the-cap filler was perfected and put into the field. Here, instead of forcing propellent into the container through the valve, the container is sealed off before capping, a vacuum is drawn on the container to remove the air and the liquid propellent under pressure is introduced into the open top. The cap is then clinched into place. This process offers unlimited choice of valves (or even solid cap closures) and allows speeds as great as with cold fill while at the same time offering all the advantages of pressure filling.

Those unfamiliar with the processes may ask what exactly is wrong with cold filling. Probably this can best be illustrated by an analogy—cold filling is to pressure filling as an arc lamp is to a fluorescent tube. Cold filling requires large quantities of energy to

chill the propellent and concentrate from ambient temperature to as low as minus forty F.—this energy is a total loss and this loss is compounded by the fact that additional large amounts of energy must be applied to the container to raise the temperature to 130°F. for the ICC test. Add to these almost wholly useless costs the waste of propellent flashing out of the containers as they are filled and it will be seen that direct waste is a serious factor.

In addition to the waste incident to operation, the cumbersome installation of compressors and heat exchangers calls for large amounts of space and mechanical confusion. Heat exchangers used for concentrates must be cleaned after each product and there is always the chance of contamination due to poor clean up. Whenever the lines are shut down there must be a recycling of propellent and concentrate through the heat exchangers or else there is the possibility that temperatures will vary which can give rise to excessive flashing and perhaps incorrect fills due to density changes. All of these disadvantages are eliminated with the pressure filling procedure since all products are handled at ambient temperatures. Piping complexity is held to a minimum in the pressure fill installation and space requirements are cut drastically.

A comparison of power requirements for the two processes reveals some very interesting figures.

Considering an ambient temperature of 70°F., it requires a removal of 20.6 BTU per pound of propellent 12 to chill it to minus 24°F. Since there are practically no systems in use where this heat is salvaged, this same number of BTU must be added to bring the temperature back to the starting point of 70°F. making a total expenditure of 41 BTU to simply introduce a pound of propellent into a can at 70°F. On this basis there will be expended 24,600 BTU per hour at a production rate of one hundred pounds of filled propellent per minute. This is the equivalent of 96 HP hrs. per hour or 72.5 kilowatt hours per hour, without considering at all the heat loss in the system.

The energy output for equivalent production with pressure fill equipment in current use would be approximately 4.0 kilowatt hours per hour.

Translated into dollars, this means that on a single shift basis there is a total waste of about four hundred dollars per month.

Added to the waste of electrical energy there is the difference in propellent loss between the two processes. On a conservative basis the cold fill process would lose at least 0.4% more based on total propellent than would the pressure fill process. Based on the output in the foregoing example this would mean a loss of propellent worth about nine hundred fifty dollars per month on a single shift basis.

When the waste of power and propellent is combined it is apparent that there is an unnecessary outlay of around seventeen thousand dollars per year for the production level in our example.

If the cold fill process offered necessarily higher quality packages, greater flexibility, lower maintenance expense, less preparation down time, more efficient use of personnel, easier to use equipment, lower first cost, or any one of these factors, it could, perhaps, be defended. The fact is that just the opposite condition prevails—the advantage lies with pressure filling on each of the enumerated counts.

Not only do the comparative overall advantages lie with pressure filling, but there is a large group of products which cannot, under any circumstances, be cold filled. These are the water base or other cold sensitive products which can not be handled in the chilled state or would be damaged by chilling. Where these products are to be considered it is necessary that pressure filling equipment be available. This being true, it is apparent that pressure filling can be universal in its application while cold fill has definite limitations.

The proponents of the cold fill process have made

Fluorocarbon propellent chart

PROPELLENT NUMBER	CHEMICAL FORMULA	CHEMICAL NAME	MOLECULAR WEIGHT	BOILING POINT, °C	UNITED STATES				ENGLAND	
					FREON	GENETRON	ISOTRON	UCON	ARCTON	ISCEON
11	CCl_3F	Trichlorofluoromethane	137.38	23.7	11	11	11	11	11 (9)	11 (131)
12	CCl_2F_2	Dichlorodifluoromethane	120.93	−29.8	12	12	12	12	12 (6)	12 (122)
13	$CClF_3$	Chlorotrifluoromethane	104.47	−81.4	13	..	..	..	..	..
14	CF_4	Tetrafluoromethane	88.01	−12.8	..	..	..	..	..	..
21	$CHCl_2F$	Dichlorofluoromethane	102.93	8.92	..	..	..	..	21 (7)	..
22	$CHClF_2$	Chlorodifluoromethane	86.48	−40.8	22	22	22	22	22 (4)	22 (112)
23	CHF_3	Trifluoromethane	70.02	−82.2	..	..	..	..	..	..
113	$CCl_2F\text{-}CClF_2$	Trichlorotrifluoroethane	187.39	47.6	113	113	113	113	113 (63)	113 (233)
114	$CClF_2\text{-}CClF_2$	Dichlorotetrafluoroethane, Sym.	170.93	3.55	114	114	114	114	114 (33)	114 (224)
114_a	$CCl_2F\text{-}CF_3$	Dichlorotetrafluoroethane, Assym.	170.93	3.2	..	..	..	..	..	..
152_a	$CH_3\text{-}CHF_2$	Difluoroethane	66.05	−24.76	DFE	152_a	..	..	..	..
142_b	$CH_3\text{-}CClF_2$	Chlorodifluoroethane	100.5	−9.4	..	142_b	..	..	..	..
C318	C_4F_8	Octafluorocyclobutane	200	−6.1	C318	..	..	..	..	..
1113	$CClF=CF_2$	Chlorotrifluoroethylene	116.48	−27.9	..	..	..	..	..	..
13B1	$CBrF_3$	Bromotrifluoromethane	148.93	−57.8	13B1	..	..	..	..	..

much of the fact that a minimum of air is left in each can as a result of the evaporative flashing of the propellent as it is filled.

It is a fact that air is effectively removed incident to cold filling, but it is also a fact that there are at least three ways of accomplishing the same thing with pressure filling.

The simplest way to remove air from a container is to make up the concentrate with a volatile ingredient (which may be a high boiling propellent fraction or a solvent) which will itself flash enough during room temperature liquid filling to displace the air from the headspace of the container. This method is especially effective for packs utilizing relatively large quantities of concentrate, although it is not necessarily limited to them.

Purging as a separate operation is another way to eliminate air from a container. This is accomplished by introducing a small amount of liquid propellent into the uncapped container after the concentrate is filled. The liquid state may be the result of a natural high boiling point or the propellent can be chilled by refrigeration. As the liquid propellent hits the surface of the concentrate, previously filled into the container, it "flashes" and displaces the headspace air. The quantities of propellent involved in such purging are in the order of one gram per can and of course the refrigeration load is insignificant. After the purging operation is completed the container is capped.

The third way to eliminate air from a container is to draw a vacuum on it after the concentrate is filled, but before capping. Obviously, capping and vacuumizing must be two parts of an integrated operation. Vacuum crimpers are supplied which will produce up to two hundred vacuumized and capped containers per minute. With proper handling of proper equipment this method is the most desirable as it is both flexible and subject to rigid control.

We have not discussed the quality control aspects of the two systems in any detail except for the matter of entrapped air. There are at least two other aspects of aerosol quality which can be influenced by the filling method. These are vagrant water content and fractionation of mixed propellents.

Vagrant water can cause a host of difficulties in aerosol packages and should generally be held to a minimum. With pressure fill equipment this can be done without difficulty. On the other hand the temperature at which cold fill operations must be carried out invariably causes accumulations of frost on the filling nozzles. Various methods of eliminating this trouble have been devised, but practically any cold fill operation will result in a quantity of frost crystals being introduced into every so many containers.

The problem of propellent fractionation is not as universally important as some other quality problems, but it can cause trouble in some formulations. In cold filling an attempt is always made to fill at as high a temperature as possible. Where propellent blends made up with very small quantities of low boiling fractions are used it is possible that relatively large quantities of it can be lost if there is an accidental temperature rise. If this happens it is possible that the functional quality of the package can suffer drastically. This condition can not exist at all with pressure filling.

As far as general quality control is concerned there is no particular advantage to either system. To date, filling accuracies of the two systems are comparable. However, there is a greater potential for improvement in accuracy where pressure filling is used since variations are dependent only on mechanical relationships rather than on a continuously changeable temperature condition.

From now on the discussion of cold fill versus hot fill will be academic. Refinements in cold filling can never overcome its inherent disadvantages. The refinements in pressure filling already in being have made it the only practical process for the future.

This chart has been prepared as a reference guide to show vital information on propellents available in the United States, England, Germany, France, Japan, Italy, Netherlands, Spain and Argentina. Information on the Russian product was, unfortunately, not available.

	GERMANY		FRANCE		JAPAN		ITALY	NETHERLANDS	SPAIN	ARGENTINA		U.S.S.R.
FRIGEN	HEYDOGEN	FRIGEDOHN	ELECTRO	FLUGENE	COL-FLON	DAIFLON	ALGOFRENE	FRESANE	FLURION	ALGEON	FRATION	ESKIMON
11	11	11	CF11	11	11	11	11 (1)	..	..	11	..	
12	12	12	CF12	12	12	12	12 (2)	12	12	12	12	
..	..	..	..	..	..	..	..	..	..	..	..	
..	..	..	..	..	..	..	..	..	..	..	..	
21	..	21	..	..	..	21	21 (5)	..	..	..	..	
22	22	22	..	..	22	22	22 (6)	..	..	..	..	
..	..	..	..	..	..	..	..	..	..	..	..	UNKNOWN
113	..	113	..	..	..	113	..	..	..	..	..	
114	..	114	..	114	..	114	..	..	..	..	..	
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Solubility of aromatic substances

By Vincent DeFeo
Dodge & Olcott, Inc.

Perfumes are made by compounding together a number of natural and synthetic aromatic substances. The propellents used in aerosols serve as excellent solvents for many perfume materials. It is imperative that perfume compounds be soluble in the propellents and concentrates: insoluble constituents can cause clogging or faulty valve operation.

It is essential that the testing of solubility be carried out over a lengthy period. The reason for this is that sometimes deposits due to incompatibility may not occur immediately, but take weeks or even months to appear.

We have tested numerous essential oils and aromatic chemicals for compatibility with the listed propellents. Our tests were carried out in clear 2-oz. aerosol bottles. Four bottles of each of the mixtures tested (listed below) were made up, two stored at room temperature, two at 110°F. One of each set was then sprayed and evaluated at one-week intervals, for approximately six months by panel of perfumers, checking for any change in odor characteristic. One set was left for solubility observations by our aerosol technicians during a sixteen month period.

1% SOLUTIONS OF ESSENTIAL OILS

Essential Oils	Propellent 11/12 (50:50)	Propellent 114
Abies Siberica	S	S
Almond Sweet Expressed	S	S
Bay	S	I
Bergamot	S	I
Cananga Native	S	I
Cassia Rectified	I	S
Cedarwood	S	S
Clove, U.S.P.	C	I
Copaiba	C	I
Eucalyptus Redistilled	C	S
Fennel, U.S.P.	S	I
Lavender Flowers, U.S.P. 40/42%	C	C
Lavandin	I	I
Lemongress Rectified	S	S
Peppermint Natural, D&O	S	S

S = Soluble C = Cloudy I = Insoluble

1% SOLUTIONS OF AROMATIC CHEMICALS

Aromatic Chemicals	Propellent 11/12 (50:50)	Propellent 114
Alcohol C-8	S	C
Alcohol C-9	S	C
Alcohol C-10	S	C
Aldehyde C-11	S	C
Aldehyde C-14	S	C
Amyl Butyrate	S	S
Benzyl Iso Eugenol	C	I
Coumarin	I	I
Geraniol	S	S
Linalyl Acetate	I	S
Methyl Benzoate	S	S
Methyl Coumarin	I	S
Musk Ambrette	S	S
Musk Ketone	S	I
Styrolyl Acetate	S	S

S = Soluble C = Cloudy I = Insoluble

Quick breaking aerosol foam

By Thomas A. Wallace, Jr.
General Chemical Division,
Allied Chemical Corporation

A host of new applications for quick-breaking-foam-type aerosols have been developed by Allied Chemical's General Chemical Division since the Company announced this aerosol innovation last April. Latest reports indicate many marketers in the toiletries, personal products and pharmaceutical fields are actively studying the new foam's advantages for their product lines.

The salient characteristic of quick breaking foam aerosols is that a small amount of product can be dispensed in a limited area, yet "goes to work" immediately by breaking into a liquid when disturbed or warmed by body heat. Thus the user is able to dispense a desired amount quickly and conveniently in a way impossible with conventional aerosol sprays and foams.

For example, when used in a nail polish remover formulation, a small drop of the new foam can be placed on each fingernail. When the foam is touched with a cleansing tissue it becomes a liquid and can be rubbed to remove the polish.

Based on our laboratory work, the five most promising new applications are as anti-perspirants, acne and other medicated foams, insect repellants, hair conditioners, and hand lotions.

Applications developed earlier and believed to be currently undergoing tests by potential marketers include after shave lotion, nail polish remover, cologne, toilet water and cold wave lotion, among others.

Basic formulation of the new aerosol foam is:

Ethyl Alcohol	46.0 - 66.0%
Surfactant	0.5 - 5.0%
Water	28.0 - 42.0%
Propellant	3.0 - 15.0%

The proportion of surfactant in quick breaking foam is thus between one-half and one-twentieth of that employed in formulations for the most popular regular foam product, aerosol shave cream.

General Chemical's technicians developed the new foam as an outgrowth of their day-to-day work in preparing new aerosol formulations for customers. About two years ago the laboratory received a request for a foamy hair tint, and last year, a customer requested an after shave foam.

Through this development work, and subsequent studies, it was found that the consistency and stability of an aerosol foam could be altered by varying the ratio of the solvents, the per cent of type of surfactant, or the type and pressure of the propellant mix.

Here are typical formulations. While the surfactant can be nonionic, anionic or cationic, it should be soluble in only one of the miscible solvents (either alcohol or water), but not in both. The particular surfactant (Polawax) used in these test formulations contributes to a clear, colorless concentrate.

Antiperspirant Foam

Formulation	Wt.%
Polawax (Croda)	1.50
SDA No. 40, anhydrous	53.20
Hexachlorophene (Sindar)	0.12
Aluminum Chlorhydrol (Reheis) (50% water soln.)	30.00
Perfume	0.18
Water, distilled	15.00
Above Concentrate 92%	
Genetron 12/114 (20:80) 8%	

To be packaged in plastic-coated glass bottles.

Acne Remedy

Formulation	Wt.%
Polawax (Croda)	1.5
Resorcin (Merck)	3.0
Salicylic Acid	1.5
SDA No. 40, anhydrous	59.0
Water, distilled (or lime water)	35.0
Above Concentrate 92%	
Genetron 12/114 (20:80) 8%	

Insect Repellent Foam

Formulation	Wt.%
Polawax (Croda)	1.5
SDA No. 40, anhydrous	45.0
Meta Delephene (n,N-diethyl toluemides) (Hercules)	18.0
Perfume	0.5
Water, distilled	35.0
Above Concentrate 92%	
Genetron 12/114 (20:80) 8%	

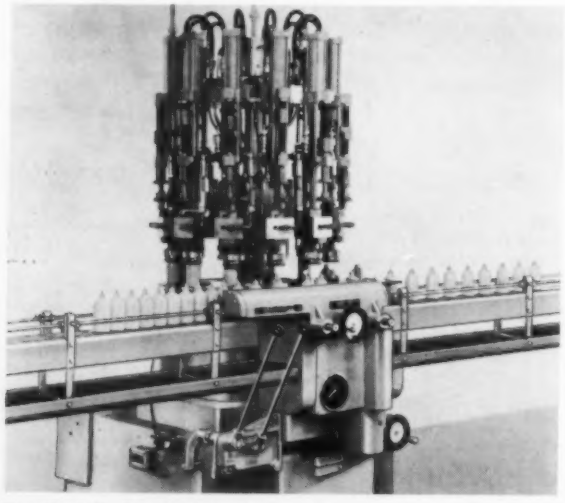
Hair Conditioner

Formulation	Wt.%
Polawax (Croda)	1.5
Solulan 16 (Amerchol Products)	3.5
Ucon 50 HB-5100 (Union Carbide)	1.0
Diisopropyl Sebacate	1.3
Cetyl Alcohol	1.0
SDA No. 40, anhydrous	52.5
Perfume	0.2
Water, distilled	39.0
Above Concentrate 92%	
Genetron 12/114 (20:80) 8%	

Protective Hand Lotion

Formulation	Wt.%
Polawax (Croda)	1.5
SDA No. 40, anhydrous	51.0
PVP/VA; E-335 (in 50% EtOH)	10.0
Solulan 16 (Amerchol Products)	1.0
Perfume	0.5
Water, distilled	36.0
Above Concentrate 92%	
Genetron 12/114 (20:80) 8%	

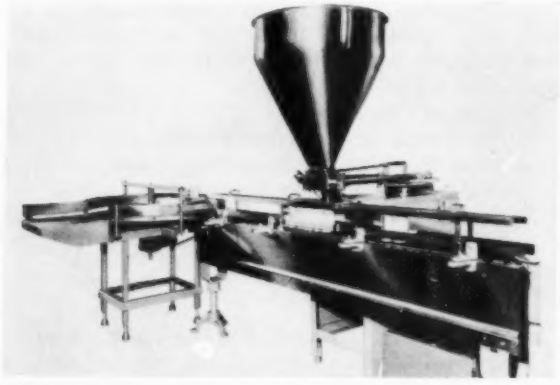
Molded nylon aerosol bottle, of duPont's Zytel resin. This bottle is pigmented with metallic gold. It is expected that this container will be approved for food product use.



The Karl Kiefer Machine Company

This firm manufactures a line of container cleaning machines, and filling machines for most types of materials that can be packed in aerosols, as well as pressure charging equipment.

The pressure charging equipment runs in size for a small two-stem manually fed unit, to entirely automatic high speed equipment up to twenty four stems on a single unit.



F. L. Burt Company

This firm has been known for many years for their filling equipment. It has been adapted to aerosol packing lines, particularly since most of the manufacturers of pressure charging equipment have tended to depend on the experience of the fillers rather than try to design comparable equipment on their own. Burt fillers are available for consistencies from liquids to very thick pastes, and in a variety of metals for every type of material.

Aerosol Valves

By A. Herzka
Pressurized Packaging
Consultants, Ltd.

Next to the propellent, the valve is probably the most important part of a pressurized pack. It is through the valve that the product is obtained and the valve determines the quantity of material which is sprayed, i.e. the rate of discharge and the spray pattern, i.e. the particle size of the spray and the pattern or shape of the spray cone.

The discharge rate which is normally expressed as grams per second, and which may be determined by a recognized method (1), is controlled in valves other than those of the metering or one-shot type, by the smallest orifices within the valve structure. This may be situated in the actuator, (also known as valve button), in the valve stem or in the valve housing. The varying location of these restrictive orifices differentiates the various makes of valves that are commercially available, and for certain products the location of these restricting orifices is very critical. As the majority of valve components are moulded in plastic it is inevitable that there should be some variation in orifice within any one type of valve and a variation in discharge rates must therefore occur. With a high quality component, however, this variation need not be more than ± 15% on a 1 gram/second discharge and 95% of all valves supplied should have discharge rates within that range. With metering valves the discharge rate is controlled solely by the size of the metering chamber.

Although the spray pattern is controlled by many factors other than valve construction as for example, the nature of the propellent, the propellent/product ratio, internal pressure and product viscosity, the valve design does play an important part, as will be described below. But once again it must be emphasized that slight variations in spray patterns are bound to occur with any one type of valve system due to the inevitable variations in the moulded components.

If the discharge rate and/or the spray pattern must not extend beyond very narrow limits then these requirements should be brought to the attention of the valve supplier in good time.

All valves consist of an actuator; of at least one further moulded or metallic component; of a housing which not only "houses" the valve components but also serves as an anchor for the diptube when the latter is fitted; of one or more rubber components and very often, a metal spring is also incorporated.

The valve assembly is mounted in a metal component, by means of which it is united with the container. In the majority of cases, this metal component consists of the well-known one inch mounting cup which may be of tinplate or aluminium, though sometimes the valve is mounted directly into one of the end components. Whenever a glass or plastic container, or a metal container with a diameter of less than one inch is utilized, then the metal component is known as a ferrule and aluminium predominates as the material. The rubber components control the effective functioning of valves and, as is well known, rubber is prone to alteration in physical form under the influence of solvents. Excessive gasket swell is the most dangerous phenomenon as it will cause the valve to fail and it is usually caused by such solvents as methylene chloride, acetone, methyl ethyl ketone and xylene. Sometimes an ingredient of the product may leach out one or more of the components of the rubber gasket and while the performance of the valve itself may be totally unaffected, the sprayed product, for instance, may be discolored. Careful pre-marketing evaluation of the valve is necessary in order to avoid such occurrences.

The solvents in a formulation can also effect other components of valves. Orifices may become distorted causing valves to leak after actuations, leading to loss of contents of the pack; dip tubes may swell and part company with the main body of the valve leading to immediate pack failure due to inability to deliver the contents. Similarly, actuators may loosen during use and while not necessarily falling off, a part of the emerging spray would be directed into the space thus created between the actuator and the valve stem, thus soiling the user's hand.

The majority of valves may be classified into broad groups, depending on the type of discharge obtained.

Standard valves are those which give a continuous emission while the actuator is operated, and as they form an integral part of the dispensers, they are discarded with the latter when the product is completely used up.

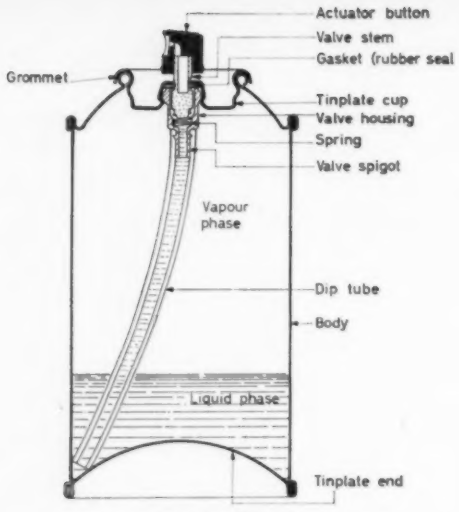


Fig.1 Valve Closed

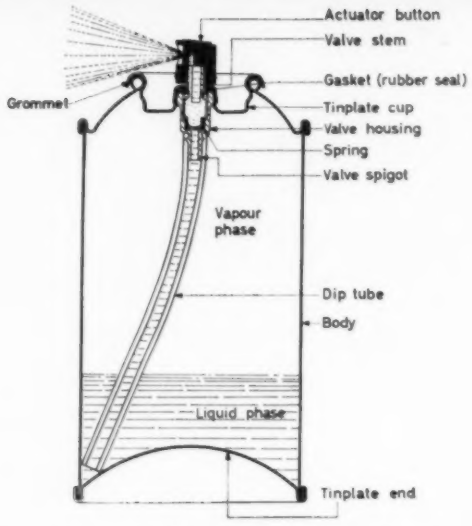


Fig. 2 Valve open

They may be divided into two sub-groups according to the nature of the actuator.

Perusal of Figure 1 indicates that the rubber gasket prevents the flow of the liquid phase (product propellent mixture) by sealing the valves stem orifice under spring tension. In Figure 2, however, depression of the actuator against the spring causes the gasket to open and to allow the passage of the liquid phase through the internal orifice into the valve stem passage and thence into the atmosphere through the external orifice.

Standard Valves with Ordinary actuators

This type of valve is the one most commonly used and is suitable for all types of space and surface sprays where liquid propellent is part of the liquid phase, providing the internal pressure of the pack is not much below 30 p.s.i.g. at 70° F. No hard and fast figure can be given because the nature of the product could easily displace the lower pressure limit in each direction.

Figure 1 shows a vertical section though one of the most popular types of valve, and this particular valve consists of a polythene actuator with the diameter of the external orifice approximately 0.02 in.; a valve stem, made of brass or nylon housing one to three internal orifices; a rubber seal; a nylon valve housing; a stainless steel spring and a dip tube (made of polythene or PVC).

Basically, the series of orifices with communicating passages act as expansion chambers. When the product-propellent (liquid) phase passes through the first orifice the magnitude of the pressure drop is such that partial boiling of the liquefied propellent occurs. This process is repeated at subsequent orifices with the vaporization of the propellent becoming more violent until complete atomization is achieved beyond the final orifice.

Standard Valves with Break-up Spray Actuators

Break-up spray actuators are necessary for two-phase systems where the liquid phase consists only of product. Such valves are also necessary for three-phase systems, other than foams, and are advisable for all other liquid sprays with a low internal pressure, which are usually packed in glass dispensers.

Atomization is achieved by forcing the product stream through a swirling chamber in the actuator (usually very close to the final orifice) which causes a turbulence in the liquid stream as it passes through the chamber to the final orifice, where it breaks up into fine particles which are, however, coarser than the particles obtained by propellent atomization.

The effort of using a break-up spray actuator instead of a standard actuator is clearly demonstrated by Figures 3A and B and 4A and B. The former illustrations represent spray patterns obtained with the formulation

65% ethyl alcohol
35% Propellent 12

with liquefied propellent present in the spray, and the latter with the formulation

66% ethyl alcohol
nitrogen at 90 p.s.i.g.

with only product and no propellent at all present in the spray.

In addition, Figures 3B and 4B clearly demonstrate that the spray particles are coarser when no propellent is present in the spray.

Foam Valves

Foam valves are primarily designed for use with aerated emissions and the actuator, or sometimes the whole valve assembly, is characterized by large internal expansion chambers which continue without interruption into the large external orifice. A product utilizing a foam actuator will yield a foam immediately after discharge from the external orifice.

A spray which gives a foam on impingement may be obtained when using a standard spray actuator other than a foam actuator. This method of dispensing is often resorted to when using glass dispensers, as very few foam actuators are suitable for use with 20 mm, or thereabouts, valve ferrules.

Stream Valves

Special valves have been developed for use in dispensing viscous material pressurized with nitrogen. These valves have considerably enlarged orifices in the valve housing spigot and the dip tube has a larger bore, both modifications being designed to permit the passage of viscous fluids. For the same reason there are multiple side orifices in the valve stem which in turn has a larger internal bore.

Fig. 3a
65% ethyl alcohol
35% Propellent 12
Valve "A"
Standard actuator

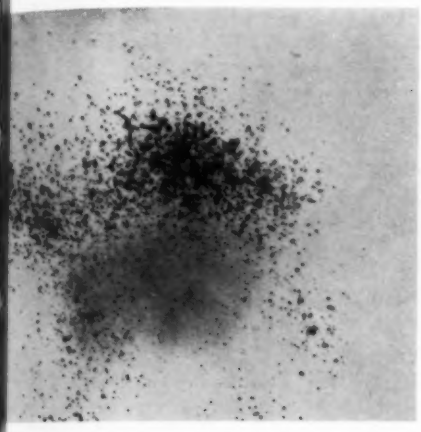


Fig. 3b
65% ethyl alcohol
35% Propellent 12
Valve "A"
Break-up spray actuator

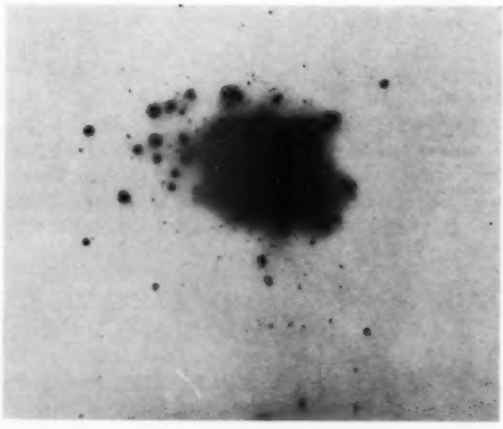


Fig. 4a
66% ethyl alcohol
nitrogen at 90 p.s.i.g.
Valve "A"
Standard actuator

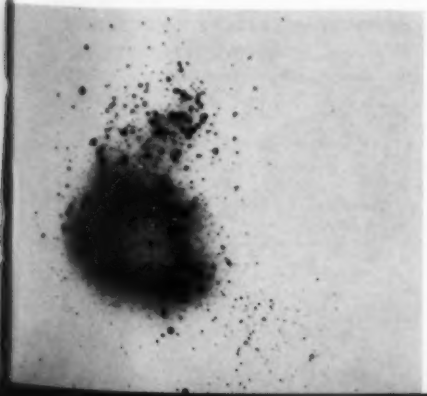


Fig. 4b
66% ethyl alcohol
nitrogen at 90 p.s.i.g.
Valve "A"
Break-up spray actuator

Metering Valves

A metering valve is one which delivers an equal dosage at each operation. Its uniqueness lies in the delivery of a predetermined quantity of material which is of definite economic interest because the administration of therapeutic dosages with pharmaceutical preparations is possible. Metering valves also find application in the packing of expensive perfumes, with insect repellants and other personal products which for reasons of cost or convenience may be packed in pocket size dispensers no bigger than ⅓ oz. The utilization of standard valves in those circumstances may well cause the dispensers to be completely emptied in an unreasonably short time.

The metering action is obtained by admitting the liquid phase (product/propellent mixture or product alone) into a metering chamber either during the up or during the down stroke of the valve and releasing it during the reverse stroke which simultaneously isolates the bulk of the liquid phase within the dispenser.

In addition, metering valves may find increasing use for packs utilizing compressed propellents. In such instances the metering device will ensure that no propellent is lost during misuse. In fact, the metering chamber may well be so large that it would not be emptied completely during a normal actuation so that the user would not even reach the cut-off stage.

One-Shot Valves

As their name implies, one-shot valves are those which discharge the whole contents of the dispensers at one time. Because the contents are discharged in one operation, usually lasting less than one minute, the valve has no other function but to seal off the contents before use and to permit the unobstructed escape of the contents during use. 'One-shot' valves are therefore of relatively simple construction and may consist of a copper discharge tube tightly sealed into the usually dished top end of the dispenser, with a sealed portion protruding outside the dispenser. The external portion is usually scored (line of weakness) and a handle engages the tube between its sealed end and the line of weakness. In order to operate the valve, the handle is pulled which causes the discharge tube to break at the line of weakness, thereby forming the orifice from which the contents emerge.

Another form of valve (Fig. 5) employs a breakable seal, situated within the dispenser. In order to operate the valve, pressure is applied on the external nylon pin causing it to push the rubber seal inwards. The mode of operation is illustrated clearly by Figures 6 (a) and (b). The same principle governs the use of another valve, fitted with an actuating button which, on operation, dislodges the disruptable seal.

Yet another type of valve has a push wire passing through the dip tube which displaces a sealing plug from the bottom end of the dip tube on operation. This plug is so constructed that it cannot re-seat itself once it has been dislodged. Most one-shot valves

are suitable for mounting in standard one-inch valve cups, with the exception of the break-off type, described above. The is only suitable for mounting in a dished end of the same diameter as the dispenser.

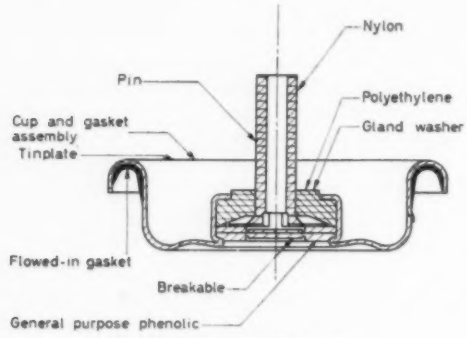


Fig. 5 A fire extinguisher valve

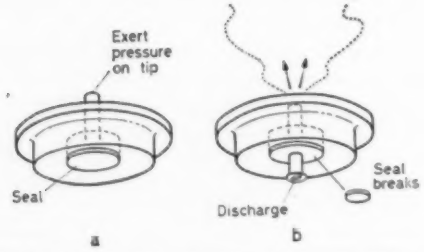


Fig. 6(a) & (b) Diagramatic representation of operating the valve shown in Fig. 5

Although one-shot valves are employed chiefly for pressurized fire extinguishers, they find use in horticulture for insecticidal sprays used in greenhouses, or for antibiotic packs, such as might be used for spraying poultry runs, beehives, etc.

Re-usable Valves

The desire to re-use either the valve or the valve and the dispenser after exhaustion of the contents, has exercised the minds of many for some time.

The only re-usable valve which appears to have found general acceptance is that designed by Galeazzi. It is generally fitted to glass dispensers which are provided with screw necks. On exhaustion of the contents, the valve is unscrewed and the dispenser is discarded. The valve, which is provided with a piercing pin, is re-used on the refill, an identical glass container, closed with an aluminium overseal which is pierced as the valve is screwed down. One of the disadvantages of this particular valve is the danger that the valve might be unscrewed (and there is no physical impediment to doing so) before the dispenser is empty.

The use of replaceable cartridges to hold propellent only has been publicized in France, but it appears to be an exceedingly expensive way of pressurizing a product, and this method one could say is probably contradictory to the very nature of pressurized packaging which aims to achieve its purpose as cheaply as possible.

Paint Valves

It has already been mentioned earlier that the rubber components of valves are prone to alteration in physical form due to the influence of solvents. This effect is particularly marked with paint packs. In addition, as is to be expected, clogging of the valve orifices and/or the actuator orifices is likely to occur in-between-use due to the drying of residual material in these areas. This is the reason why paint packs often bear the legend "After spraying, invert dispenser and give a short burst". The aim of this is to clear spray residues from essential orifices by means of pure propellent from the head space. Nevertheless, the selection of a suitable valve should take into account that directions for use are not necessarily obeyed by the public. The valves that are most popular for paint packs are those where the salient orifices are in the actuator, so that in the event of a blockage which can not be cleared by pricking the orifice with a pin, or by soaking the actuator in a solvent such as turpentine, the actuator itself may be replaced without difficulty. With other valves, themselves very suitable for non-paint packs, the salient orifices are often inaccessible and blockages therefore cause pack failure.

Powder Valves

With powders the problems are fairly similar to paints in respect of valve blockage, though swelling of rubber components is not generally met with. The problem is not so much a dry film as a matter of impactation of dry particles in and around the orifices and valve passages. The valves that are commonly used for paints are also the most satisfactory ones for powders.

Miscellaneous Valves

Amongst the miscellaneous valves which have been developed is one which permits only product to be expelled during operation, irrespective of the manner in which the dispenser is held, i.e. the dispenser may even be held upside down. One of the valve manufacturers has achieved this by fitting a slide to the dip tube, this slide covering a porthole in the dip tube when the dispenser is held upright. On inverting the dispenser, the slide uncovers the porthole thus admitting product to the dip tube. Another manufacturer has achieved a similar effect by drilling a relatively large hole into the valve housing.

A number of adaptors are also available which may be fitted to the conventional actuators in order to achieve a special product distribution. Flexible tubes for delivering woodworm killer into flight holes and special adaptors for asthma sprays are examples, as are also eye drop applicators.

Method of Injecting Propellent

Injection of propellant through the valve is possible in virtually every type described above, with the exception of the one-shot valves. Even metering valves can be filled by injection.

For one-shot valves, the cold filling or under-cup filling are the only possibilities.

References:

1. Herzka. A. & Pickthall, J. **Pressurized Packaging (Aerosols)** 2nd Edition 153 (1960) (Butterworths, London)

Questions and answers on aerosol packaging

The editors have asked the following two experts in their fields several questions concerning some aspects of aerosol packaging that seldom receive open attention. We believe that their frank answers are of genuine interest.

The following two questions were answered by A. R. Marks of the Wheaton Plasti-cote Corporation.

Q. *What are the advantages of the non-adhered coating versus the bonded coating?*

A. The non-adhered bottle coating utilizes the physical extensibility of the plastic jacket to relieve the pressure integral in an aerosol package through vent holes deliberately placed in the jacket at the time of manufacturing and retain the glass fragments within the jacket. A broken bottle is immediately detectable.

During our early development work on plastic coated aerosol bottles, adhesive coatings were the only materials available. The noted disadvantage was that we were not utilizing the full elongation of the plastic and there was no method of releasing pressure except by accidental perforation. Since accidental perforation was unreliable and extremely dangerous, a search was instituted for a non-adhesive material. The adhesive materials were then abandoned. The non-adhesive materials in use by Wheaton today take advantage of the natural elastic qualities of the Polyvinyl coating which has an expansion factor of 400 to 500 percent elongation.

The positive and controlled venting of the jacket, a patented feature of the Wheaton package, insures a release of rapidly expanding gases and prevents rupture of the coating.

Q. *Would you hazard a guess as to how many coated glass aerosol have exploded in consumers use to date? Do you know of any reports of harm resulting to users as a result of such an accident?*

A. In seven years of marketing coated containers, or over 300 million units in consumer use, to our knowledge there has never been an accident resulting in a bodily injury claim.

We have been called to investigate approximately six cases presumed to be accidental and hazardous breakage. In each of these six cases there was marked evidence of deliberate attempts to fracture the container. Scientific examination revealed eight to fourteen severe impact marks similar to hammer blows upon the container.

No claims resulted in any of these cases since evidence was conclusive of deliberate attempts to fracture package. We firmly believe that the margins of safety are more than adequate to insure a non hazardous package provided the manufacturers recommendations are observed.

The following two questions were answered by L. G. Cannella of Continental Can Company, Incorporated.

Q. *Why are there so few aerosol shampoos and antiperspirants in metal containers?*

A. Shampoos and antiperspirants are products which are known in the metal container industry as "hard-to-hold" products, and prior to the introduction of aerosols, were not packaged in metal containers because of their hard-to-hold properties. Until quite recently, very little work was done to re-formulate commercial shampoos and antiperspirants for the purpose of packing them in aerosols.

Taken individually, shampoos are sold by well-known companies under well advertised brand names in unusual and identifying packages. Any attempt to change these well-known and accepted packages involves many considerations on the part of the merchandisers. We are convinced that after the due "considerations" have been made, shampoos in aerosols will be marketed.

In addition to being a "hard-to-hold" product in metal, antiperspirants are generally sold in cream form. Cream is not the most adaptable type product for aerosol dispersion, but antiperspirants are now on the market in aerosols and we predict will soon be seen in ever increasing numbers.

Q. *Wouldn't the soldered aerosol container present more opportunities for leakage than the seamless drawn container?*

A. To answer this question properly, several premises should be established, such as . . . . What is this container expected to do? . . . . How much pressure is it expected to hold? . . . . What type seamless container is referred to, a seamless bottom can, or a seamless top can—as well as, I assume, seamless body? . . . . Are price considerations involved?

The container known as the aerosol container is made to comply with specific Government regulations. It is not intended to hold high-pressure compressed gases. Side seams of 3-piece aerosols are more than adequate to hold pressures required under Government regulations, as attested by the fact that over twenty billion have been used since their inception. Aerosol style containers in which greater pressures are developed than permitted under ICC laws will buckle at the bottom on 3-piece cans, not at the side seam. Seamless top containers would also buckle at the bottom under the same pressures, and a seamless top container would buckle at the top. Certainly a seamless container, including the bottom, would hold greater pressures than a seamed body with a seamed bottom, or seamless body container with a seamed bottom. If the special requirement of extremely high pressures is needed, a completely seamless can should be used, assuming that the added cost is not an important factor. Such a seamless can was the original aerosol used during World War II to supply our men in arms with individual insecticide containers.

PACKAGING & PROMOTION

1

1—Allen B. Wrisley

Allen B. Wrisley has introduced Superbe Bubbling Bath Oil in gold-decorated, milk opal glass decanters. Available in three fragrances the product creates bubbles and a water-softening action eliminating bathtub ring. The product will retail at $1.00 a bottle.

2

2—Guerlain

L'Heure Bleue, a Guerlain fragrance, is now available in bath and hand size bars of soap. The cake is curved to fit the hand and is made in a shade of blue that gives the bar a translucent quality. The wrap is silver foil and Guerlain paper with antique design and lettering. It will be available in boxes of three bath-size or three hand-size cakes.

3

3—Helene Pessl

Helene Pessl has introduced a new display unit for "Little Lady" Lip Treats. The washable plastic unit uses a three-dimensional concept in displaying the product in projected slots with embossed lettering and a raised top panel for brand indentification. The case is longer, and slimmer and features a ridged, swivel base. The Lip Treats are available in two flavors, cherry and strawberry and the deceptive red shade is used to appeal to children.

4—Dunbar Laboratories

"Atrix", a silicone hand cream developed in Europe, is being marketed here by Dunbar Laboratories in a polyethylene tube. The stand-on-cap tube is lined to prevent loss of oils from within. An external barrier coating provides protection against oxygen transmission and gives a glass like sheen to the package. The product is available in 1⅛ ounce tubes and 3 ounce tubes.

4

5

6

5—Corday

Slated for national distribution before the holidays are two of Corday's newest products packed together in a gift box. "The Five Hundred" an aerosol containing 500 metered sprays of Corday's Concentree Cologne and a refillable purse-size Spray Parfum containing 1½ drams of Corday perfume. Called a "Spraygrance Duo", the package will sell for $7.50 and will be available in the company's Toujours Moi and Fame fragrances only.

7

6—Faberge

Faberge "Esprit de Parfum" is an aerosol container which has no cap. The container is made of silver plate and holds over 800 metered sprays. Aphrodisia, Woodhue, Tigress, Flambeau and F# are available in this new container. The spray, packaged in a gift box, will be sold for $5.50.

7—Shulton

Shulton's Spray Colognes are now packed in sculptured-gold, stained-glass aerosol containers. Promoted as a decorator touch for the boudoir and a conversation piece each bottle will have its own gift carton. The Spray Colognes are priced at $2.00 for 2 ozs.

8

8—Stanley Home Products

An aerosol hair dressing for men has been brought out by Stanley Home Products, Inc. The product is a creamy liquid and is called "Creme Hair Dressing". A nitrogen propellent is used in the aerosol to act as a "pusher" rather than combine with the material to make a foam. The dressing is part of Stanley's "Chairman" line of men's good grooming aids. The suggested retail price is $1.00.

Book Reviews

KOSMETOLOGIE, by J. Stephen Jellinek. Dr. Alfred Huthig Verlag, Heidelberg, Germany. 624 pages, 1959, Price 40 DM (approx. $10.00).

The European cosmetic literature is always refreshing because it is written from a different point of view than our own. However, this often results in exceedingly mediocre publications. Indeed, it has not been since Winter and Cerbelaud that anything truly worthwhile has been published abroad.

Now comes Jellinek, a chemist perfumer, son of a renowned perfumer, with his survey of the present status of cosmetic science. The book is divided into two parts. The first deals with principles underlying the function and manufacture of cosmetics. The second part deals with the solution of cosmetic problems including formulations.

The author refers extensively to the world literature bringing together under one cover a vast digest of present day science and practice. Although patents are widely mentioned, the formulation sections in particular are a bit weak in this respect.

A comment must be made on the proofreading. A number of misspelled words have slipped past the author, Arocel for Arlacel (p. 378), Pieckthall for Pickthall (p. 214), Cosmetocology for Cosmeticology (p. 260 and several other places), Lantarol for Lantrol (p. 368) are a few examples.

The product formulas are all quoted from the cosmetic literature. None seem to have originated from the author, but he makes no pretense at originality in this respect.

The book is exceedingly up to date and will be one of the best foreign language cosmetic books for a long time.

The author is unusual in his organization and presentation of material. The publisher has done a commendable job of printing and binding. You will want this book for the "latest word" on almost any given cosmetic subject.—(M. G. de N.)

REMOVING SPOTS AND STAINS. I. and E. Mellan, Chemical Publishing Co., Inc., New York 10, New York. 95 pages, 1959, Price $4.50.

The authors have prepared an useful basic book on spot and stain removal. Although intended for professional cleaners, it is useful for the homemaker.

Nine chapters describe fabrics, dyes, rules for stain removal, Equipment, stain removing agents and methods of using them.

While seemingly high priced, this small booklet will find many uses. —(M. G. deN)

ELEMENTS OF ION EXCHANGE. Robert Kunin, Reinhold Publishing Corp., New York 22, New York. 173 pages, 1960, Price $5.75.

This is another of a series of small and inexpensive books on various new subjects. It is intended for those with little or no theoretical background.

The book covers the broad aspects of ion exchange in nine chapters and three appendices. It is clearly written. The text is well arranged and easy to understand. —(M. G. deN)

DICTIONARY OF ECONOMIC PLANTS. J. C. Th. Uphof, Hafner Publishing Co., New York 3, New York, 400 pages, 1959, Price $9.75.

The author, an emeritus professor of botany, catalogues alphabetically some 6000 different species of plants. The first plant mentioned is Abbevillae Fenzliana Berg., while the last entry is Zygophyllum simplex L. Each entry lists the scientific and common name, geographical distribution, products derived and commercial use, if any.

The book fills a need felt by all who have dealt with botanical products and materials derived from them. —(M. G. deN.)

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BUILDER'S NEW "Combination Drill Press Capper" for Aerosol Bottles and Cans has a Production Rate of approximately 2 per minute with Aerosol Bottles . . . 8 per minute with Aerosol Cans.
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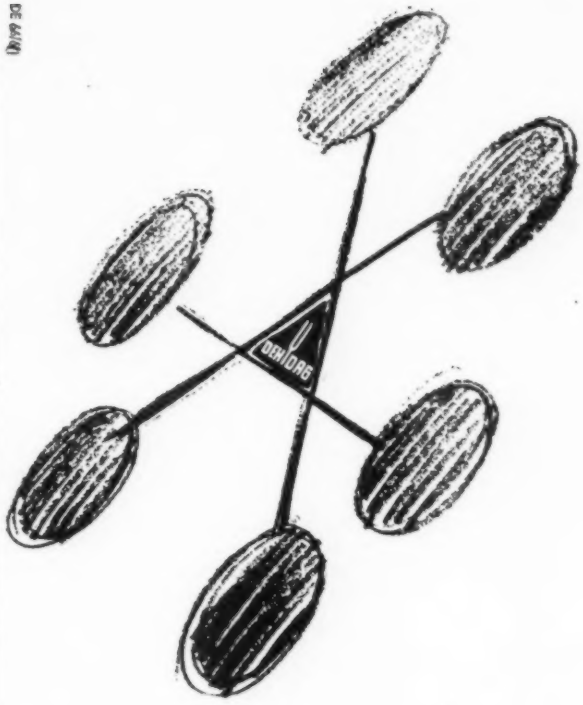
TECHNICAL ABSTRACTS

Soaps.

Soap compositions in various physical forms containing phenolic bactericides were color- and odor-stabilized by adding minor amounts of azines, nonaromatic hydrazides, or hydrazonium salts of organic acids (Olin Mathieson Chemical Corporation, Brit. 802,447). Deterioration of odor can be retarded by using certain hydrazides, incorporated during any stage of manufacture (Unilever Ltd., Brit. 795,674). The many problems involving the perfuming of detergent products were reviewed (Fourman, Soap Chem. Specialties, 35 (8), 43-45, 102-103). A fresh natural odor for toilet soap may be achieved by using 0.02 to 1.5% by weight of dihydroterpinyl acetate (Fiore, Kitchens and Saunders, U. S. 2,889,254). A soap composition contained germicidal amounts of the reaction product of silver chloride and ethylene thio-urea (Baldwin, U. S. 2,864,768). In contrast, the silver salt of an aromatic sulfinic acid has been used (Lutz and Sharpe, U. S. 2,864,769). Addition of 10 to 20% quaternary ammonium compound to soaps prevented hard water precipitation and was apparently compatible (Aylesworth, U. S. 2,861,-955). Soap may be protected from insoluble salt formation by using 1 part of a fatty acid-protein condensation product and about 3.5 parts of condensed sodium phosphates. All are kneaded to form a tablet (Chemische Fabrik Grunau Akt.-Ges., Ger. 922,432). Soaps may be formed in place by mixing 45% of yellow grease (containing at least 5% free fatty acids), 8 to 9% caustic soda, 25% of a 51% sodium silicate solution, and 21 to 22% water softeners such as phosphates, carbonates, and silicates. These are mixed and covered with the saponifiable material (Thurman, U. S. 2,861,953) ready for subsequent usage. *Thru J. Am. Oil Chemists' Soc., 37, 238 (1960).*

The Influence of the Manufacturing Method and of the Hydrogen Ion Concentration upon the Viscosity of the Guar-gum Mucilages (Der Einfluss der Herstellungsmethode und der Wasserstoffionenkonzentration auf die Viskositat von Guar Gummi-Schleimen) by Rosmarie Bolliger. (Pharmaz. Forschungsabteilung F. Hoffmann-La Roche AG, Basel, Switzerland.)

Guar gum consists mainly of guaran, a nonionic, highly polymeric poly-saccharide consisting of mannose-chains with galactose as side-chains. Guar mucilages give pseudoplastic (quasiviscose) flowing curves. The obtainable values of viscosity are very much dependent upon the method of production. Obviously the meal of the guar seeds contains enzymes which are capable of splitting the polygalactomannose chains and thus causing a loss of viscosity. The enzymes can be destroyed by heating the aqueous unbuffered mucilages for 30 minutes at 100°C. without damaging the polysaccharides. Heated mucilages show higher values of viscosity and a better stability of viscosity than the nonheated ones. As in other mucilages



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the initial wetting of the guargum with nonsolvents (ethanol, glycerin, polyoles) is advantageous for the hydration, but their addition can be effected only up to a certain quantity because otherwise dehydration sets in. The highest viscosity values are reached in the pH values 5-8, regardless of whether or not they are fixed with hydrochloric acid, sodium hydroxide or phosphate buffers. Minimum viscosity is found at the pH values 3 and 10. The viscosity values of the sufficiently preserved and de-enzymatized guar mucilages remain fairly constant after they are fully hydrated at a pH optimum of 5-7 in buffered, and of 6-8 in unbuffered solutions. This fact is especially remarkable as the mucilages of natural polysaccharides generally are more inclined to a depolymerization. *Thru Am. Journal of Hospital Pharmacy, 17, 311 (1960).*

Fat Emulsions. The Oxidation of Fats in Emulsions by Per Finholt. (Pharmazeutisches Institut, Universitat, Oslo, Norway.

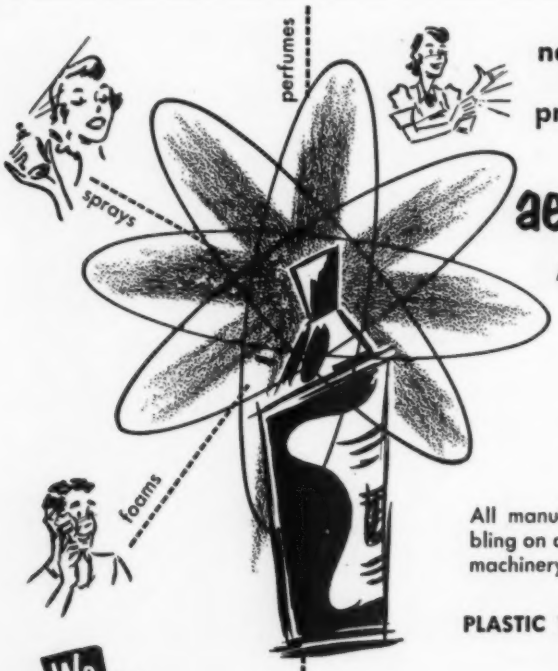
It is the objective of this work to examine the following problems: 1. How quickly does the fat oxidation occur in emulsions? 2. Which factors influence the speed of oxidation? In order to be able to follow the oxidation of the fats the author worked out a method for the quantitative evaluation of peroxides in emulsions. The method is a modification of the well known method by Wheeler. The influence of the water contents as well as that of the eventual air contents of the emulsion in the determination of peroxides was examined. Comparable tests on the speed of oxidation in the following systems were effected: fat alone, fat and emulgator, fat and emulgator and water in the form of an emulsion. Some emulsifiers speed up the oxidation of fat (pro-oxidative effect), others hinder it (anti-oxidative effect). To be able to answer the question if perhaps the pro-oxidative effect is being caused by the copper contents of the emulsifiers, a method was devised to quantitatively determine the copper in emulsifiers and in fats. The author tested whether or not the relation between copper contents and pro-oxidative effect of emulsifiers was completely clear. Further the pro-oxidative effect of copper in emulsions was studied whereby the copper was introduced either as copper oleate to the fat phase or as copper sulfate to the aqueous phase. *Thru Am. J. Hosp. Pharm., 17, 313 (1960).*

Masked Incompatibilities. "Masked" Incompatibilities in External Remedies by H. v. Czetsch-Lindenwald (Pharmazeutische Fakultat, Universitat, Alexandrien, V.A.R.).

The author coined the expression "masked" for all those incompatibilities which by themselves are not externally perceptible, but in which nevertheless certain forces, generally those of chemico-physical nature, diminish or even destroy the therapeutical effect. In external remedies and also in suppositories an increased importance has to be attributed to processes which may act in this sense, mentioned in the following:

1. Adsorptions:
   (a) by adsorbing pigments, such as kaolin, bentonite, aerosil;
   (b) by water-soluble colloids, such as alginates, gums CMC and CM;
   (c) by lipophilic parts, such as the external phase of 2/o emulsions.

Continued on page 103



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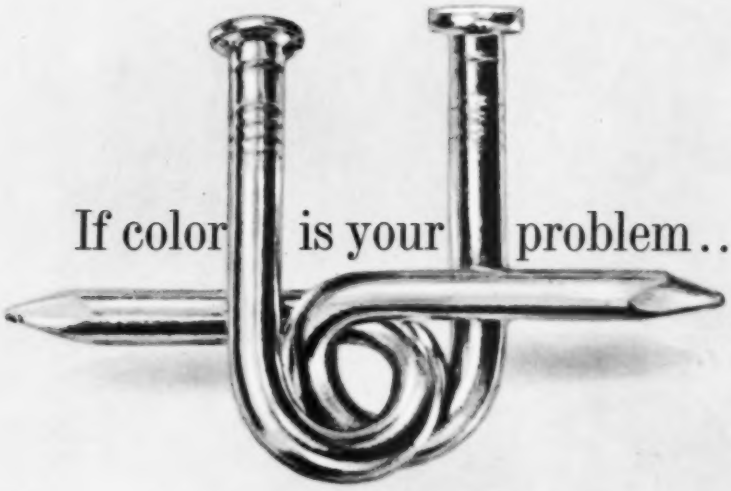

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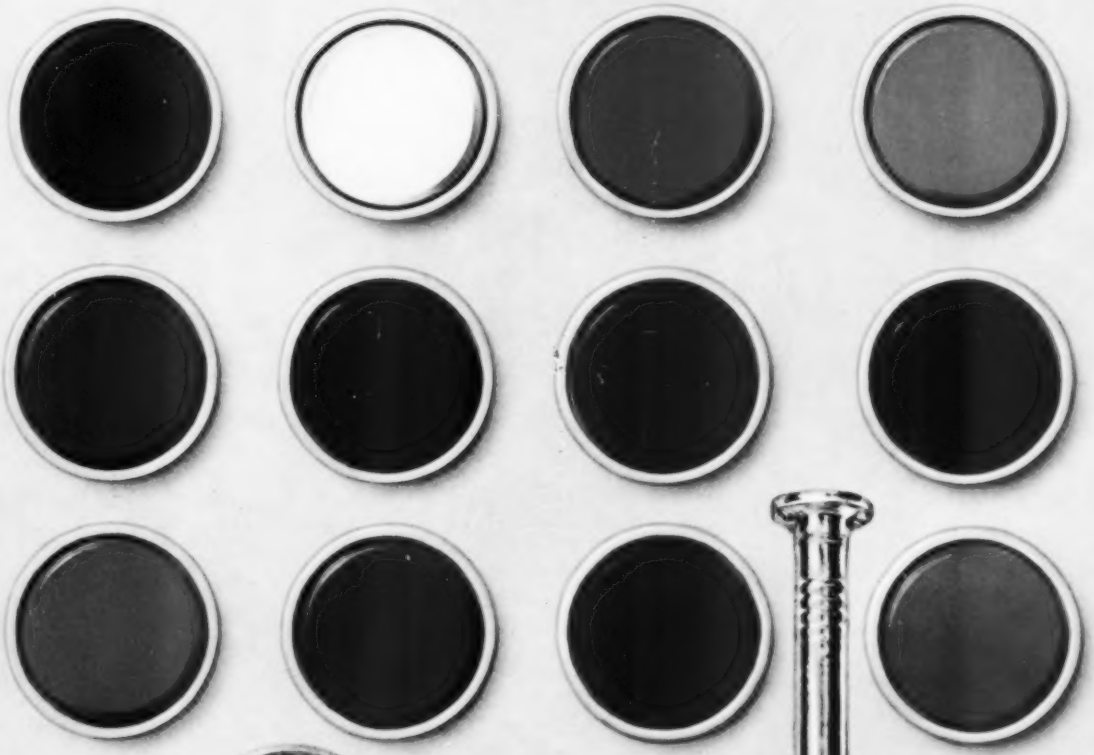

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Continued from page 96

2. By the absence or by an incorrectly arranged solution gradient.
3. By an osmotic stream which is opposed to the resorption pathways.
4. By hidden physical processes:
   (a) the "walling-up phenomenon"
   (b) the formation of semipermeable membranes;
   (c) the affect of wetting agents.

By means of experiments it was demonstrated that every single group mentioned above challenges the therapeutical results. Thus for instance, (1) a powder in which the fat is bound by an adsorbens does not fatten, (2) an ointment (w/o emulsion type) in which the water-soluble active substance has been absorbed by the fat, is probably non-effective and (3) a suppository of this very same kind is certainly not effective at all. The absence of the solution gradient in the case of ointments, water in nature, is also of decisive importance. Thus for instance, an effective substance particularly well soluble in polyglycol ointments, but poorly soluble in water, will remain in the ointment and has no chance to diffuse into the skin. The "walling-up phenomenon" appears then, when an active substance is surrounded by the drug carrier in such a manner that this active substance cannot diffuse towards the outside. Wetting agents draw on the skin and may interfere with the effect of the ointment. In addition, they change the Ca-, Al-, and Mg- stearates, which usually do not adsorb due to their hydrophobic character, into strong adsorbents. The report concludes with the request to pay increased attention to such "masked" incompatibilities between the different components and the auxiliary mediums. *Thru Am. J. Hosp. Pharm., 17, 313 (1960).*

A Note On Autoxidation and Its Inhibition in Wool Alcohols B.P., E. W. Clark and G. F. Kitchen, from Westbrook Lanolin Company, Argonaut Works, Laisterdyke, Bradford 4, Received December 1, 1959

The effects on Wool Alcohols B.P. of gradual autoxidation during storage at room temperature have been compared with the known effects of accelerated oxidation. There is a decrease in emulsification value, a bleaching of the surface, and, contrary to previous reports, a rapid fall in melting point. Autoxidation, even under bad conditions, has been inhibited, apart from a slight fall in melting point, for at least 23 months by the addition of 500 p.p.m. of butylated hydroxyanisole, but certain other antioxidant systems had only a limited effect. *Thru J. Pharm. Pharmacol. 12, 4, 233(1960).*

STATEMENT REQUIRED BY THE ACT OF AUGUST 24, 1912, AS AMENDED BY THE ACTS OF MARCH 3, 1933, JULY 2, 1946 AND JUNE 11, 1960 (74 STAT. 208) SHOWING THE OWNERSHIP, MANAGEMENT, AND CIRCULATION OF

American Perfumer published monthly at Pontiac, Illinois for October, 1960.

1. The names and addresses of the publisher, editor, managing editor, and business managers are: Publisher, Mrs. E. R. Allured, 418 North Austin Blvd., Oak Park, Ill.; Editor, Stanley E. Allured, 418 North Austin Blvd., Oak Park, Ill.; Managing editor, none; Business manager, James W. Allured, 418 North Austin Blvd., Oak Park, Ill.
2. The owner is: Allured Publishing Corporation, 418 N. Austin Blvd., Oak Park, Ill.; James W. Allured, 418 N. Austin Blvd., Oak Park, Ill.; Stanley E. Allured, 418 N. Austin Blvd., Oak Park, Ill.; Allen R. Allured, 418 N. Austin Blvd., Oak Park, Ill.
3. The known bondholders, mortgagees, and other security holders owning or holding 1 percent or more of total amount of bonds, mortgages, or other securities are: None.
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JAMES W. ALLURED, Business Manager

Sworn to and subscribed before me this 23rd day of September, 1960.

[SEAL] FRANCES L. PATTERSON

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WRITE FOR SAMPLES AND FULL INFORMATION

Antibacterial Synergism Between the Halogenated Bisphenols and the Halogenated Aromatic Anilides and Carbanilides. D. R. Noel, R. E. Casely, W. M. Linfield, and L. A. Harriman (Armour & Co., Chicago, Ill.). Appl. Microbiol. 8, 1-4 (1960).

It has been demonstrated that some of the halogenated bisphenols show antibacterial synergism when combined with some halogenated aromatic anilides and carbanilides by tests in vitro. This synergism was found to exist for the chemicals, per se, and to be independent of the carrier used, whether water, soap, nonionic or anionic (other than soap) synthetic detergent. The most active synergism was found for the higher halogenated bisphenols, that is, the hexachloro- and tetrachloro-compounds, with the trichlorocarbanilides and the chlorinated salicylanilides *Thru J. Am. Oil Chemists' Soc., 37, 210 (1960).*

Sulphurized Oils III: Sulphurizing of Linseed Oil With S_2Cl_2 and Sulphur. G. Schiemann, H. During, and F. Vonwinkel (Inst., Tech. Chem., Hannover). Fette Seifen Anstrichmittel, 61, 913-917 (1959).

In continuation of the series of publications on the subject, the authors give the details of manufacturing conditions and application characteristics of sulphurized oils, which are prepared with the aid of S_2Cl_2 and elementary sulphur. The oils obtained are compared with one another, especially concerning the change brought about in their properties through the replacement of S_2Cl_2 by sulphur. The sweallability and the resistance of the sulphurized products to alkali and ultraviolet radiation is also discussed. *Thru J. Am. Oil Chemists' Soc., 37, 200 (1960).*

Vitamin A Stability. On the Stability of Vitamin A in Ointments by F. Dal Brollo, G. Polasek and S. Rigamonti (Farmitalia-Stabilimento, Milan, Italy).

To test the stability of vitamin A in ointments, three ointment bases which are often pharmaceutically employed were manufactured. The ointment bases correspond to the following three main groups: water-free, water containing and "Oil in water."

(a) Base No. 1—water free:

Rx.	White petrolatum	23.7
	Liquid petrolatum	10.0
	Eutanol[R]	10.0
	Amphocerin[R]	50.0

(b) Base No. 2—water containing:

Rx.	Sodium alginate	3.2
	Glycerin	10.0
	Distilled water	75.5

(c) Base No. 3—"oil in water":

Rx. Lanetta N^{R}	15.0
Spermaceti	10.0
Liquid petrolatum	5.0
Glycerin	10.0
Distilled water	53.7

Parallel to the above mentioned series of bases a further series of ointments were prepared, which differed from the first only by the addition of antioxidants, chosen on the basis of earlier experience—a mixture of 0.05% butylhydroxyanisol with 0.05% ascorbylpalmitate was used as stabilizing agents. For the tests, as far as the vitamin A is concerned, the palmitate was chosen; it was added to the base in a concentration of 50,000 I.U. vitamin A to one gram of ointment. The various ointments were packed in tin tubes. Part of them were stored at room temperature, the other part in a thermostat set at 40°C. At regular intervals the contents of vitamin A were tested by analysis. The results of the analytical controls are mentioned and the resulting conclusions are discussed. *Thru Am. J. Hosp. Pharm., 17, 313 (1960).*

The Definition and Classification of Waxes. C. Ludecke (Dervio, Italy). Fette Seifen Anstrichmittel, 61, 999-1001 (1959).

The author deals with the modern definition of waxes which has been published in the Official Methods of the DGF. This definition takes into consideration their technological properties only but does not take their origin and chemical constitution into account. To supplement his postulations on the classifications he draws from the work of F. Gieser. The allotment of the waxes of berries and fruits in the classification is explained. *Thru J. Am. Oil Chemists' Soc., 37, 200 (1960).*

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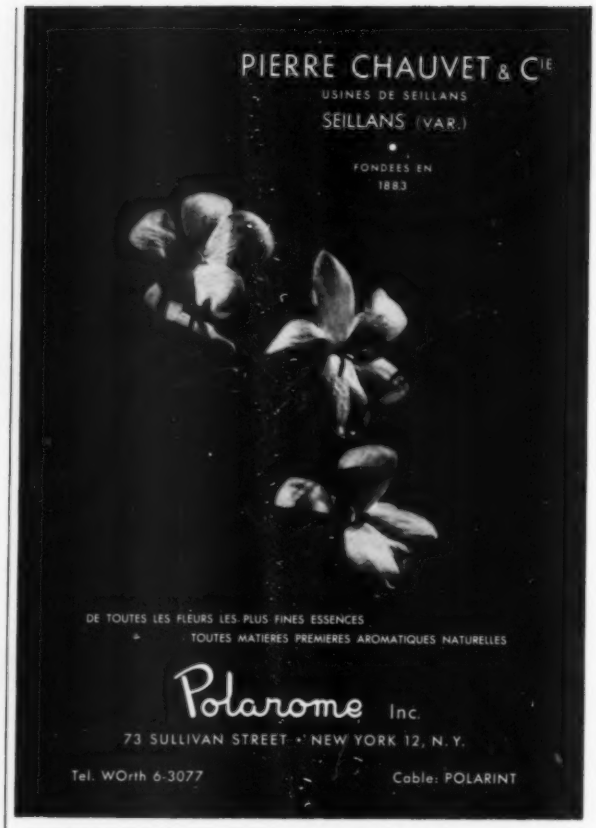

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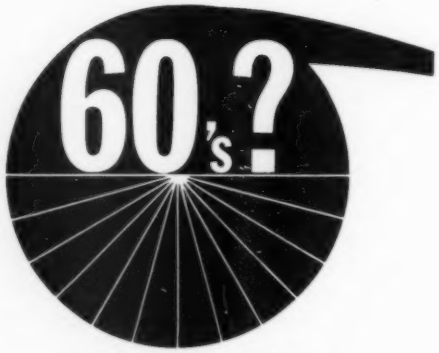

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BRANCHES THROUGHOUT THE WORLD

. . NEWS AND EVENTS . . . NEWS AND

Day is president of DCAT

James Day, New York District Manager of The Dow Chemical Company, was elected President of the Drug, Chemical and Allied Trades Association (A Section of the New York Board of Trade) at the Association's Annual three-day meeting at the Sagamore Hotel, Lake George, N. Y.

James Day

Named to serve with him were: Wm. J. Schieffelin, III, V. P., Schieffelin & Co., as Vice President; F. M. Schwemmer, Pres., Pharmaco, Incorporated, as Treasurer; William J. Quinn, Merck & Co., Inc., as counsel; and Helen L. Booth, as secretary.

NBBMA 1961 convention to be in NYC February 23-25

The 1961 Convention of the National Beauty & Barber Manufacturers' Association will be at the Statler Hilton Hotel, commencing Thursday afternoon, February 23, 1961 through Saturday noon, February 25, 1961.

Warren Freeman, of Clairol, Incorporated, is again convention chairman.

Rubinstein honored

Helena Rubinstein, Inc. was honored by the Medical Health Center of America in recognition of the firm's services in advancing the psychiatric cause of the Denver Institution. This is a free, nation-wide, nonsectarian psychiatric treatment and research center devoted to the care of the borderline metally ill.

Max Factor announced record sales for first half 1960

Max Factor & Co. sales for the first six months ended June 30, 1960 amounted to $27,269,464, a new record high. The corresponding period of 1959 saw sales totalling $24,453,676 and net profits of $1,770,121. The 1960 first half net profits were $1,638,214. Unusual expenses of a non-recurring character were blamed for the decrease in earnings. Heavy promotion expenses and costs in moving manufacturing, warehousing and shipping facilities were among the expenses absorbed during this period, according to Max Factor, Jr., president.

Cinderella International Co. bought by Dumas Milner Corp.

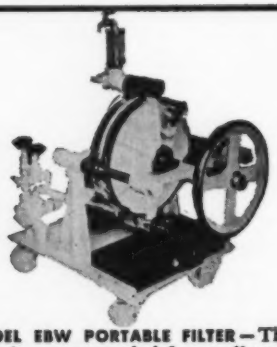
The Dumas Milner Corporation, has reported its purchase of controlling interest in Cinderella International Co., Inc., Orlando, Florida, a retail marketing organization for household products and an extensive cosmetic line sold under the Cinderella name.

Cinderella products are presently sold in twenty-two states via the "party plan" method of distribution, by over 2,000 retail representatives.

Meredith E. Dobry, founder of Cinderella International, will continue

Cosmetic Career Women, Inc., luncheon in September. On the dias, left to right: Miriam Baumgarten, Publicker Alcohol & Chemical Sales: Centa Isermann, President, Van Dyk & Co., guest speaker, who discussed "Chemistry & Cosmetics—A Great Team"; Eve Henriksen, Dana Perfumes Corp. luncheon chairman; June Clark, Macfadden Publications, and Dolle Condon, Ungerer & Co.

FILTERS AND BOTTLE FILLERS
for the Perfumer



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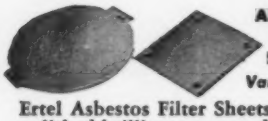

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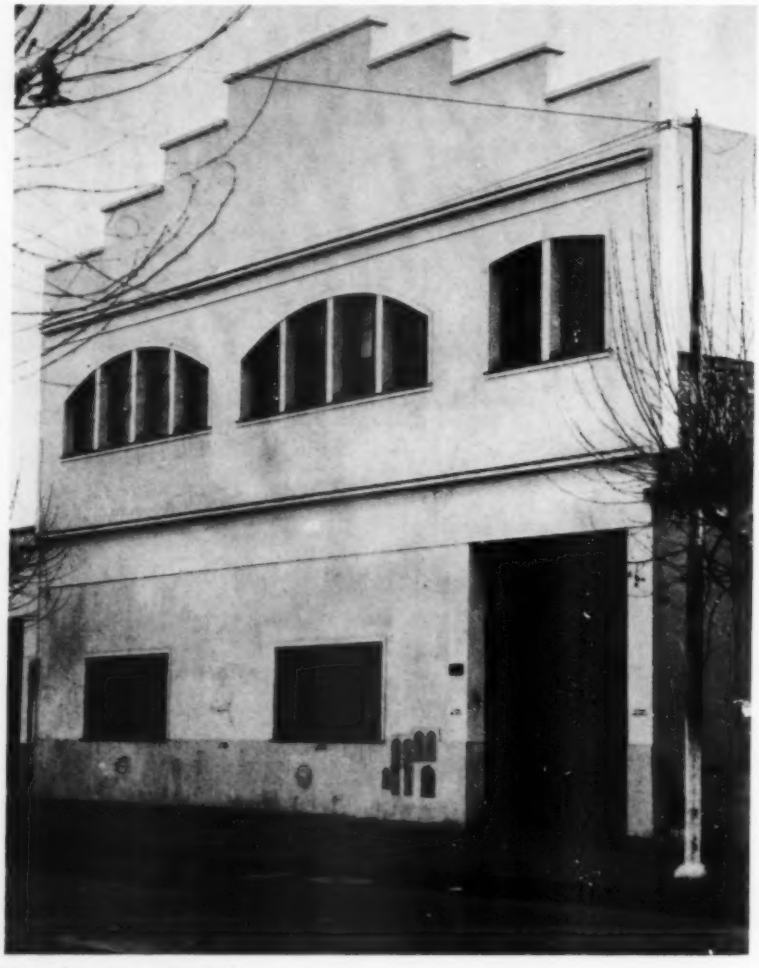
with the company as vice president in charge of sales. Howard S. Cohoon has been named president and Vincent E. Tateo is executive vice president of the expanded Cinderella operations.

ATI adds large volume filling line

With the installation of an entirely new line including a 12-head vacuum crimper and a 12-head high-speed rotary pressure filler, Aerosol Techniques, Inc. has increased its manufacturing facilities more than 50%. The new line operates at a speed up to 150 units a minute, handling all types of products.

Liquefied petroleum gassing is to comprise a major role for the new line as ATI steps up its LP program following extensive research and successful application by the company's California affiliate, Western Filling Corporation.

Along with the cold-filling lines ATI will be thoroughly tooled for the production of not only two-phase systems but the aqueous-based products which represent an increasingly important aerosol area.



Givaudan Argentina S. A.

Pilot plant production has just been started in the new factory of Givaudan Argentina S. A., according to Mr. Leon Givaudan, president of the newest manufacturing member of the international Givaudan organization.

Givaudan Argentina S. A. was organized in 1955 for the purpose of serving the expanding soap, perfume and cosmetic industries of the Argentine.

In the village of Bernal, a suburb of Buenos Aires, compounding and research operations are already being conducted in a modern building owned by the company. A new plant will be located in the town of Gutierreza, twenty miles from the Argentine capital, on the River Las Conchitas. On a plot of land, some thirty acres in size, the production facilities are being planned for development and expansion.

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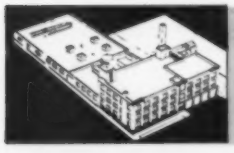
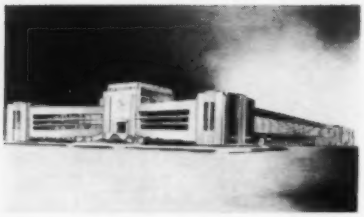


ACRES OF AEROSOL PACKAGING FACILITIES



Fleuroma moves to new plant on Long Island

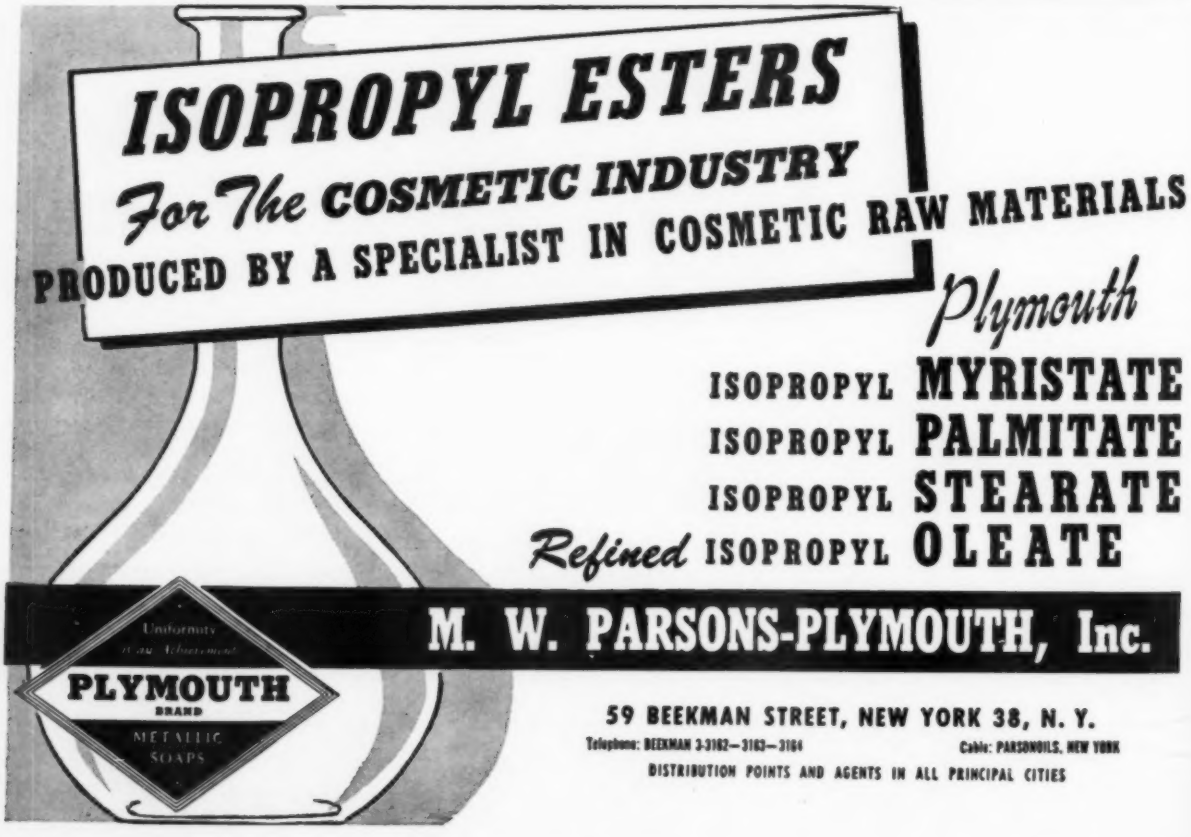
Fleuroma, Inc., has moved to new plant and offices at 43-23 Dreyer Avenue, Long Island City 1, New York. The new telephone is Ravenswood 1-8282.

Extensive renovations in this building have fitted it for modern perfume laboratories and aromatic chemical manufacturing.

High-density poly to take larger share of cosmetic packaging

Containers of high-density polyethylene plastic are moving into a more prominent position in cosmetic packaging, due to the high-density qualities of rigidity, better barrier properties, better chemical resistance over conventional polyethylene. For most products, an interior coating, commonly required for conventional polyethylene, is not required with the high-density type.

On tour with Society of Cosmetic Chemists headed for the First International Congress of Societies of Cosmetic Chemists in Munich, Germany were (above) M. G. deNavarre, first president of the International Federation of SCC, Mrs. de Navarre, J. H. Amsterdam, president of SCC of U.S.A., and Mrs. Amsterdam. Below left Mr. Amsterdam presents a gift to Kurt Pfeiffer, president of the Swiss SCC, on behalf of the U.S. tour members and to the right he presents a gift to Dr. Ludwig Masch.



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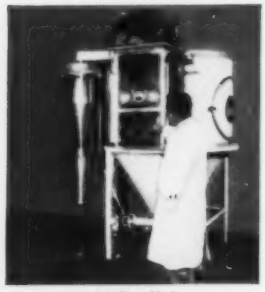
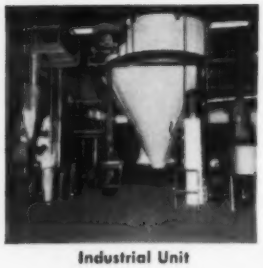


Utility Unit

Pilot Unit



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cussion of the costs of today's drugs in light of the high costs of research and the cost of living in general. "Prescription for Tomorrow" also traces the growth of medical-pharmaceutical research and, by using specific examples from Smith Kline & French's laboratories, illustrates the complex nature of this research.

Under the guidance of Dr. Robert Haakenson, SK&F manager of Community Education, head of the program, the Speakers Bureau swelled its ranks to its present 260 speakers. For the most part, these speakers are volunteers from the professional service department of SK&F and many of them are active in the Toastmasters, or the groups they address—Lions, Kiwanis, Rotary, Elks.

Naarden opens its training institute

In order to assist in meeting the future demand for trained specialists, the "Naarden" Chemical Factory, Holland, decided to open its own training institute for specialists in the chemical field.

Instruction is provided by qualified teachers and courses lead to recognized diplomas. Thirty students have enrolled for the initial course; they are divided into three groups: chemists, technical chemists and analysts.

Should the experiment succeed, "Naarden" will introduce courses in other branches of the industry in the new institute, which is known as "Naarden Technicum".

P&S International N. V., now to be IFF (Nederland) N. V.

Since its merger with Van Ameringen-Haebler last year, Polak & Schwarz International N. V. has been trading under its former name. Now, however, it is changing its name to conform with the parent organization, and will henceforth be known as International Flavors & Fragrances I.F.F. (Nederland) N. V.

Obituary

Leslie S. Beggs died September 12. He served as president of the Styron-Beggs Company from 1936 until 1958, when he retired. He was an officer and member of the board, of the Flavoring Extract Manufacturers Association almost continuously from 1936 until 1957.

Mark M. Biddison, 68, former president of Allied Chemical's General Chemical Division, died Sept. 18th at Sarasota, Florida where he lived since his retirement in 1957.

Mr. Biddison, whose career with General Chemical spanned 39 years, was president from 1952 to 1955.

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LOS ANGELES SAN FRANCISCO PORTLAND, ORE.

The officers and directors of the American Society of Perfumers met in Middletown, N. Y., as guests of Bernard Polak, president of the society, to discuss preparations for the Society's fall activities. Sitting (left to right) Dr. Anthony Frascati (By-Laws), Mrs. Dorothy Douglass, Secretary, Dr. Oliver L. Marton, Board Chairman. Standing, (left to right) Mr. Bernard Polak, President, Mr. John Hancock, Director, Mr. Herbert Sommer, 2nd Vice-President, Mr. Edward J. Shuster, Director.

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Society of Cosmetic Chemists of Great Britain, 1960 summer conference

The 1960 Summer Conference of the Society of Cosmetic Chemists of Great Britain was held on Thursday, 25th of August 1960 at the Royal Society of Arts, London. The Conference was attended by about 130 participants, most of whom had met at a cocktail party the night before.

The Society's president, Dr. H. W. Hibbott, took the chair for the morning session. In opening the Conference the president welcomed those attending and extended an especial welcome to the American Society's members present including their president, Mr. Amsterdam and to other overseas visitors.

The first paper, entitled "Surface chemical techniques in the design of cosmetic preparations" was summarised by its author, Dr. A. M. Posner. Slides were shown of a typical surface tension-concentration curve for n-butyl alcohol and also a surface pressure-area curve for the same alcohol. In the subsequent discussion mention was made of the use of natural gums as emulsifiers and of the effect on an emulsion of the addition of ethyl alcohol to the water phase. It emerged from the discussion that the stability of an emulsion cannot yet be forecast on theoretical grounds alone.

The second morning paper was on "The techniques of foam measurement" by Mr. G. E. Neu was summarised by the author. Seven different measurements were described for shampoo and correlated with these measurements. Photomicrographs and films of foams were shown.

Following this came an American paper: "The resistance of acetylated lanoline derivatives to hydrolysis" by Messrs. L. I. Conrad, K. Motiuk and H. F. Maso. The paper was summarised by Mr. Conrad. The resistance of acetylated lanoline derivatives to hydrolysis is of importance in their use in cosmetic emulsions. It appears that the drop in pH of emulsions containing these bodies was no greater than that of control emulsions, presumably, therefore, the extent of hydrolysis was negligible.

The final paper of the morning session was "Methods for determination of monoester content of propylene glycol monostearate non self emulsifying". This paper was written by Dr. C. H. Johns and Mr. W. P. Pepper and presented by Mr. W. P. Pepper. The authors had found that both acetylation and chromotographic were improvements on the periodic acid oxidation method and are

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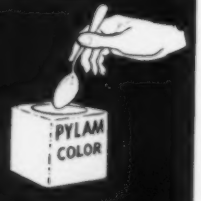

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deserving of more exhaustive examination.

Dr. R. H. Marriott, immediate past president of the Society, opened the afternoon session by expressing regret that Mr. J. Pickthall was unable to present his paper in person as he was at present in South Africa. The paper titled "The musk odour" was briefly summarised by Mr. Butterfield and dealt with the main chemical groups in which some members had been found to possess a musk odour, namely substituted benzene tetralin, indane, nitro-benzene, steroid and macrocyclic compounds.

The second paper on the laboratory evaluation of new packaging materials was presented by Messrs. S. J. Palling and E. J. Macaree. Slides were shown of the apparatus used in bonding plastic materials and also testing the resultant seals and sachets. The speaker stressed the importance of systematic testing as applied to the complete pack and the value of actual transport testing by road, rail or post, sufficient individual packs used for each test.

After a short interval for tea the company reassembled for the presentation of a paper "Care of the skin with particular reference to climate and season" by Dr. W. Schneider of the Stadtische Hautlinik, Augsburg, Germany. In his paper Dr. Schneider discussed the sensitivity of the skin in relation to the seasons of the year, clearly showing the differing reactions of dry and greasy skins to winter and summer conditions. Slides were shown illustrating the varying efficacy of sunscreens according to the different methods of application, whether in emulsion, simple solution or Aerosol.

The last paper was presented by Dr. H. W. Hibbott and Mrs. J. Monks. "The preservation of emulsions-p-hydroxy benzoic acid ester partition coefficients." It dealt with the determination of the solubility of methyl p-hydroxy benzoate in various organic materials commonly used in cosmetic emulsion formulation. He related these solubilities with the corresponding partition coefficients of the methyl ester between the respective organic substance and water.

Appreciation on behalf of the Americans present was expressed by their Society's president, Mr. Amsterdam.

The Conference day ended with an informal dinner held at the Connaught Rooms. This was an extremely pleasant end to an extremely pleasant day; the Society is much indebted to Mr. A. Herzka who organised both.

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PERSONALITIES

Leo J. Amorosi

Leo J. Amorosi has been appointed director of the Essential Oil and Aromatic Divisions of the Norda Essential Oil and Chemical Company, Inc. Mr. Amorosi, a native New Yorker, joined Norda in 1944 after graduating from Adelphi Academy and St. John's University.

W. Brian McLarnon has been appointed assistant manager of distribution and promotion for Smith Kline & French Laboratories. In his new position, McLarnon is responsible for the development and direction of SK&F's distribution programs and activities. He reports directly to Frazier Cheston, director of distribution.

Antoine de Laire arrived in the United States on October 17. He plans to spend about a month in the United States visiting his friends and calling on customers as well as attending business and directors' meetings of De Laire, Inc.

Lehn & Fink Division has announced the following promotions and personnel appointments.

Roger M. Kirk, Jr., has been promoted to the position of assistant general manager and will be responsible for the directing and coordinating of marketing activities of all products of the Division with the exception of Young Look cosmetics.

Samuel F. Melcher, Jr., has been advanced to the post of merchandise manager. He will now be responsible for supervising and coordinating the brand planning, advertising and promotion activities of all divisional products.

William S. Watchman has been promoted to the position of assistant brand manager. He will be responsible for brand planning activities on Etiquet and Lysette, and will assist Mr. Melcher on Lysol.

John Jennings, formerly vice president in charge of marketing for Noreen, Inc., recently acquired by Lehn & Fink Products Corporation, has been appointed marketing manager for Noreen products.

Henry L. Ford

Henry L. Ford has been elected president of Eastman Chemical Products, Inc., succeeding Dr. L. K. Eilers, who becomes vice chairman of the board of directors.

Ford, a vice president of both of these Kodak divisions since 1958, began his association with Tennessee Eastman in 1922. He has been closely associated with the development of the various groups connected with the sale of the products of Tennessee Eastman and Texas Eastman.

Philip J. Wagner has joined the staff of the Robinson-Wagner Company Inc.

E. R. Durrer

Mr. E. R. Durrer, president of The Givaudan Corporation, has just returned from an extended European trip during which he visited the Givaudan plants abroad. He reported that the economy in Switzerland and in Europe in general is maintaining its vigorous pace, and that Givaudan plants abroad continue to operate in high gear.

Jeanne Richards has been appointed national home sales manager for Dorothy Lamour, Inc. Miss Richards, formerly with Seligman & Katz, cosmetics firm, will supervise house to house sales of Dorothy Lamour cosmetics.

David H. Kutner has been appointed director of marketing of the Products and Lentheric-Kings Men divisions of Helene Curtis Industries, Inc. In this newly created position, Kutner will supervise the coordination of product development and research, merchandising, advertising and sales.

George Fuller, head of the Colgate-Palmolive Company's perfumery and essential oils section, has returned to the United States after a four-week European trip to investigate the production and processing of Spike Lavender and Lavandin.

Thomas R. Francis, has been assigned to the New York branch Glass Container Division of Owens-Illinois, as manager of glass container sales to the drug and chemical industries.

Carl A. Setterstrom, has been named vice president of marketing for the Rexall Chemical Company. He was formerly general manager of marketing for AviSun Corporation.

Robert A. Armstrong has joined the Harshaw Chemical Company as assistant sales manager, Fine Chemicals. A member of SAACI and a member and past president of the Cosmetic Industry Buyers & Suppliers, Mr. Armstrong was formerly associated with the Goldschmidt Chemical Corp.

Joseph J. Raispis

Garland G. Corey has been appointed to the research and development department of the Colgate-Palmolive Company's Household Products Division. Mr. Corey joined the cleansers and specialty products research section at the Colgate-Palmolive laboratories in Jersey City.

Nils S. Dahl, general sales manager of the John T. Stanley Co., Inc. has accepted the appointment as chairman of the Soap Division of Travelers Aid Society of New York's 55th Annual Fund Drive. The Society's corps of devoted volunteers and staff, stationed in the terminals and on the piers of New York, helped 60,600 travelers during 1959.

Joseph J. Raispis and **Jack W. Little** have been appointed to the development and service department of Emery Industries, Inc. This reflects an expansion of the technical service section in anticipation of Emery's new ozone oxidation facility which soon will be on stream. Mr. Raispis will provide service for Emery's lines of azelaic acid, dimer acids, pelargonic acid, and synthetic lubricant esters, while Mr. Little will handle technical service for the company's line of Plastolein Plasticizers, and Metholene fatty esters.

James P. Measday has been elevated from general sales manager to marketing director of Ivers-Lee Company. As marketing director for the world's largest unit-packaging producer, Measday will be responsible for the formulation of overall policy in connection with the company's sales, merchandising, advertising and public relations programs.

William L. Martin, Jr., has been appointed assistant manager, Glass Container Sales Administration, and Assistant Manager, Sales Development for the Food Industry, in the Packaging Materials Division of the Armstrong Cork Company.

Jack W. Little

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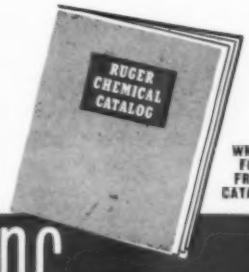

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Mr. Robert Mendoza, vice president of Antoine Chiris, has just returned from a visit to the European operations of the Antoine Chiris Company in Tadworth, England and Grasse, France. While in France, Mr. Mendoza visited various plantations and distilleries operated by the Antoine Chiris Company who are actively engaged at the moment in distilling their Lavender and Lavandin crops.

Milton Rosenberg

Mr. Milton Rosenberg has joined Geigy Industrial Chemicals Division, customer service department as product manager in charge of surfactants. Mr. Rosenberg attended Virginia Polytechnic Institute; he comes to Geigy from Gulf Research.

Dr. M. M. Gladstone

Dr. M. M. Gladstone has been promoted to manager of the Emcol Division of Witco Chemical Company's Organic Chemicals Division. In his new post, Dr. Gladstone is in charge of Emcol marketing, new product development, and coordination of production and sales.

R. M. Szymanski has been appointed manager of the Bay Minette, Alabama Plant of Newport Industries Company, a Division of Heyden Newport Chemical Corporation. He joined Newport Industries in 1960 after prior experience with Olin-Mathieson Corporation, E. I. DuPont de Nemours and Columbia National Corporation.

Robert A. Mohrman has been named manager of pharmaceutical sales for Commercial Solvents Corporation. He joined Commercial Solvents in 1953 as a sales representative for the Industrial Chemicals Department. In 1958 he was appointed assistant to sales manager of the Department.

Frank Kennedy has been appointed head of the Microbiological Division of Leberco Laboratories, Roselle Park, New Jersey. He has a Masters Degree in bacteriology from Bucknell University and was formerly the supervisor of microbiology at White Laboratories, Kenilworth, New Jersey. Also newly appointed to the staff are Shirley Schwartz, who has a B.S. in bacteriology from Cornell University and Joan Wilk. Ruth Fiuczynski has been appointed to the endocrinology group.

Dr. John H. Pearson has been appointed director of advance research planning for Allied Chemical's General Chemical Division. With the division 24 years, Dr. Pearson has served in a number of key technical capacities including those of assistant technical director, a post he held for eight years, and director of laboratory research.

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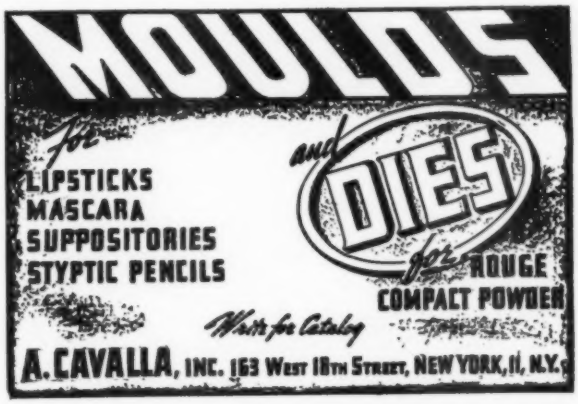

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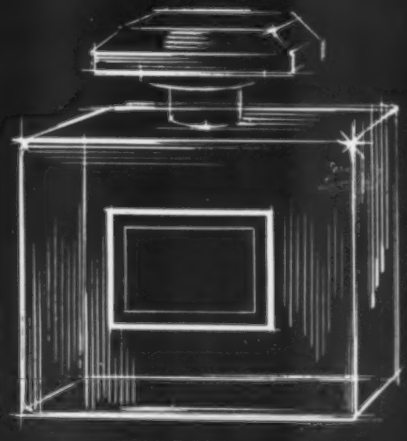

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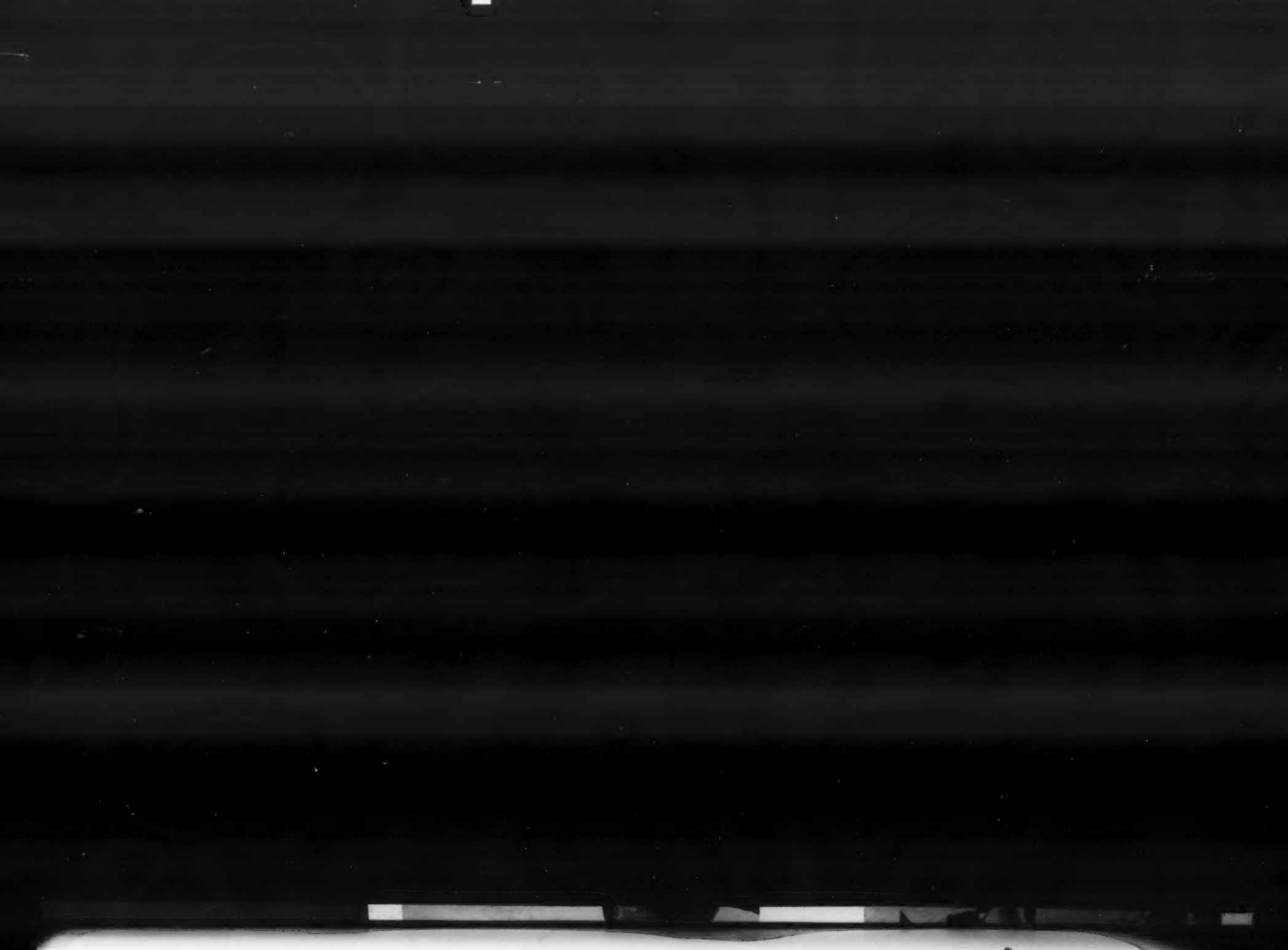
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